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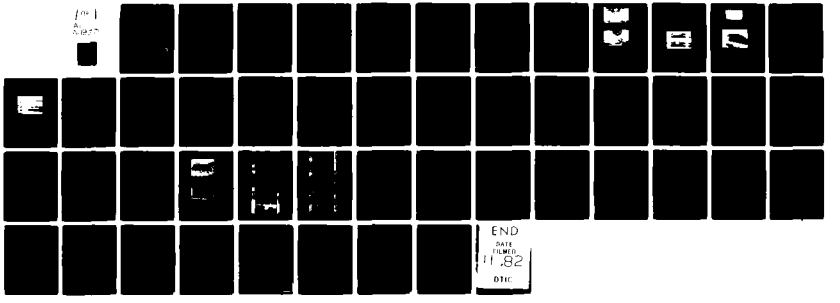

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Report N00014-82-C-0286

A LOW RESISTANCE INFRARED BOLOMETER FOR USE WITH A SQUID DETECTION SYSTEM

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Low resistance thin film bolometers for infrared detection when impedance matched to a SQUID amplifier were fabricated from a range of gold/germanium alloys (12 to 25 atomic per cent gold). The low resistances of 0.1 to 0.2 ohms, including contact resistances, were obtained in the direction perpendicular to the film plane. High resistance versions with conduction parallel to the film plane were fabricated simultaneously. The resistance for the parallel conduction was characteristic of a semiconductor, but for the

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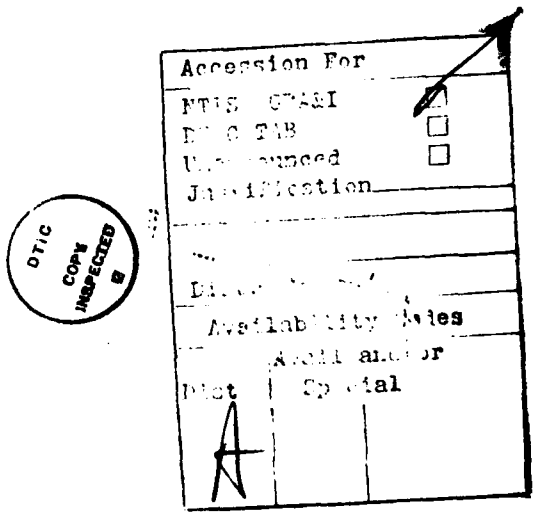

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Summary

Low resistance thin film bolometers for infrared detection when impedance matched to a SQUID amplifier were fabricated from a range of gold/germanium alloys (12 to 25 atomic per cent gold). The low resistances of 0.1 to 0.2 ohms, including contact resistances,were obtained in the direction perpendicular to the film plane. High resistance versions with conduction parallel to the film plane were fabricated simultaneously. The resistance for the parallel conduction was characteristic of a semiconductor, but for the perpendicular mode it was characteristic of a metal. Available data suggests that the metallic conduction was caused by uncontrolled gold particle sizes which could equal the thickness of the film.

Introduction

Bolometry is an attractive infrared detection technique for many purposes. It is a broad band, medium speed, high sensitivity method which has been developing for many years in the fields of astronomy and surveilance. At present, bolometers for near-ambient IR detection are cooled to the range of 0.3K to 10K, depending on the application and electronics, in order to reduce background noise. The two main types of bolometers being developed at present are high resistance bolometers with the highest possible change in resistance with temperature, and superconducting bolometers which depend on the transition edges of metals as they change from the normal to the superconducting state. The high resistance bolometers are frequently used with cooled FET-type amplifiers, while the transition edge bolometers must be used with SQUID amplifiers. The best performances reported to date have been about equal for these general types.

Each of these types of bolometers suffers some deficiencies, however. The high resistance bolometers have higher sensitivities, the higher their resistance. Therefore, noise and amplifier input impedances can become problems, along with ancillary technology such as the difficulty of making electrical contact to them. The transition edge bolometers suffer from a limited dynamic range and are difficult to make and operate.

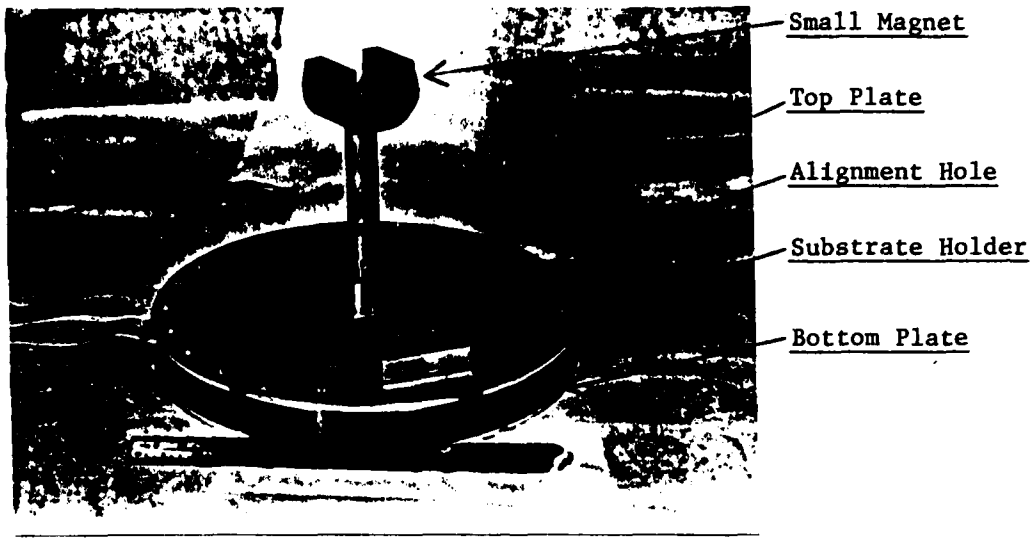
Therefore, for this contract the development of a low resistance bolometer was proposed which could be impedance-matched to a SQUID amplifier to give a low noise, high dynamic range bolometer detector. Since the speed of response and noise characteristics of a bolometer are directly dependent on the heat capacity of the sensor, a natural candidate was a thin film sensor on a low heat capacity substrate. Such thin film sensors were being made at Lake Shore for the high resistance mode (longitudinal conduction) from a range of gold-germanium alloys that had very favorable temperature-resistance characteristics and low dependence on magnetic fields.

Calculations showed that if scaling of resistance and sensitivity were linear in going from the high resistance longitudinal mode to the low resistance transverse mode, a state-of-the-art bolometer could be made. After this demonstration, other materials could be examined for still higher sensitivities to advance the art, along with integration into signal processing systems.

Experiment

A range of gold-germanium alloys were made with 12 to 25 atomic % Au in quarts tubes connected to vacuum system at about 8×10^{-7} Torr by a rubber hose. The alloy was melted by an acetylene torch and agitated by hand until judged thoroughly mixed. It was cooled by freezing it in a spiral around the inside of the tube so that it could conveniently be broken into small pieces. The alloys were evaporated in a turbo-pumped twelve-inch bell jar system at about 15 cm distance from a tungsten spiral filament in a 10^{-6} Torr Vacuum. The evaporation was done through a set of three stainless steel shadow masks to define the geometry. The mask changer and masks are shown in Fig. 1 and explained in the caption. A second sapphire substrate and set of masks were in the changer at the same time to form an array of four-lead resistors in the longitudinal conduction mode for test purposes. Such an array, and a detail of a single resistor, are shown in Fig. 2.

In addition to the separate resistor array, two longitudinal resistance test strips with eight voltage taps each were formed at the ends of the bolometer substrate in order to determine contact resistances by the method of Shockley [1], and to provide correlation of the longitudinal film properties with those of the transverse bolometers. An overall view of a bolometer and a detail of the layered structure are provided in Fig. 3. Step coverage and alignment of the various layers were the chief concerns, and in these respects the changer worked well. The layers were tapered on the edges due to the angles of the substrates to the filaments; thus by proper choice of filament and substrate positions good step coverage by successive layers could be assured.



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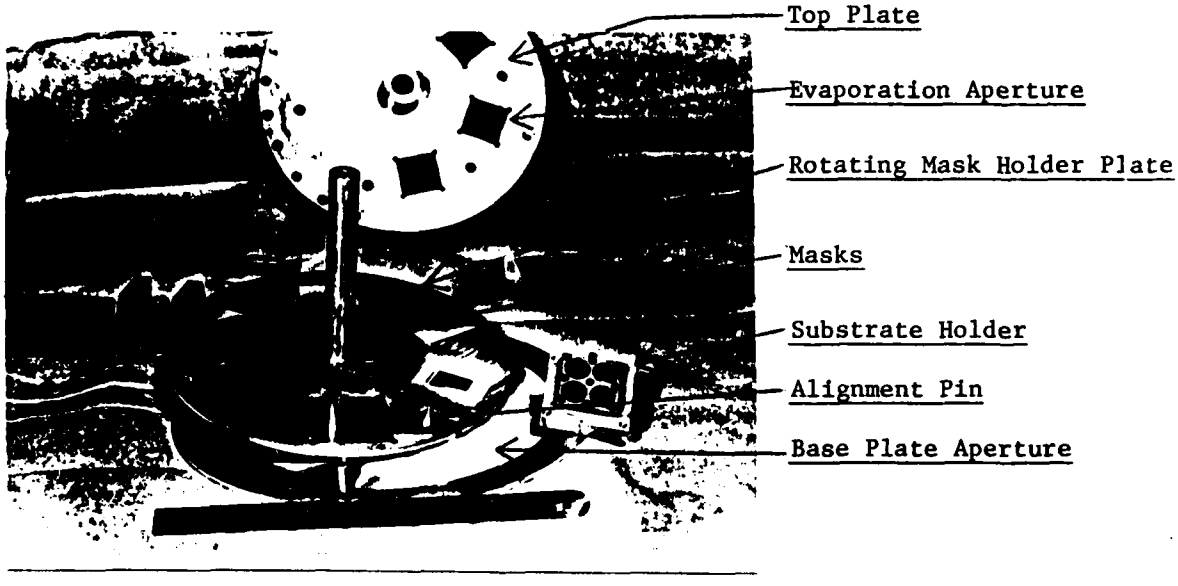
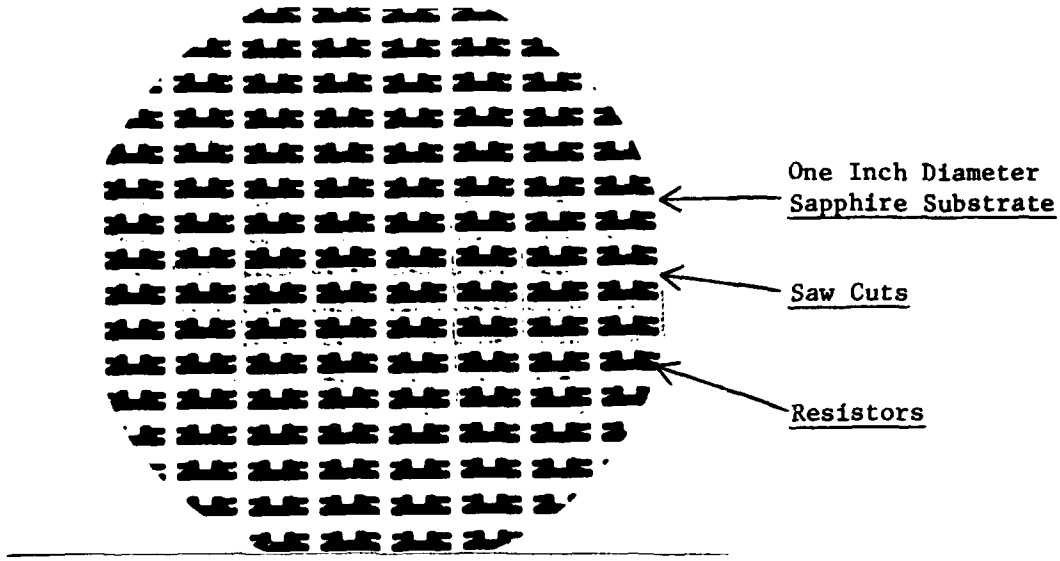


(b)

Fig. 1. Rotating shadow mask changer for use in a 12 inch bell jar. (a) The changer is rotated using a large magnet outside the bell jar, operating upon the small magnet. By bringing the large magnet down upon the bell jar, the rotating mask holder is drawn up against the top plate and into contact (if desired) with the substrate. An alignment pin on the rotating portion (b) fits into one of six holes on the top plate. The masks and substrates are waxed into the fixture with Apiezon "W" wax.



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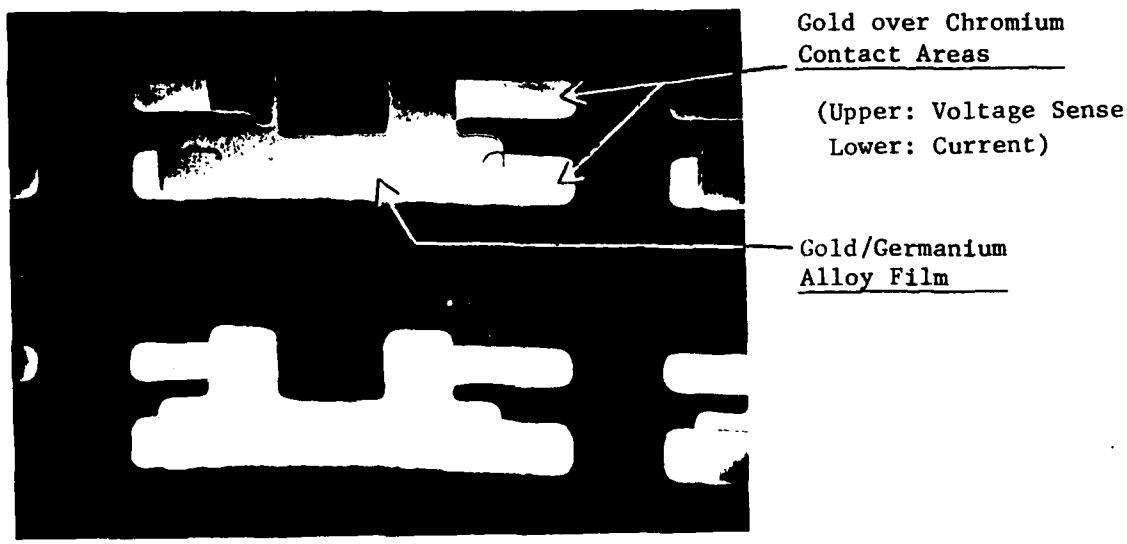
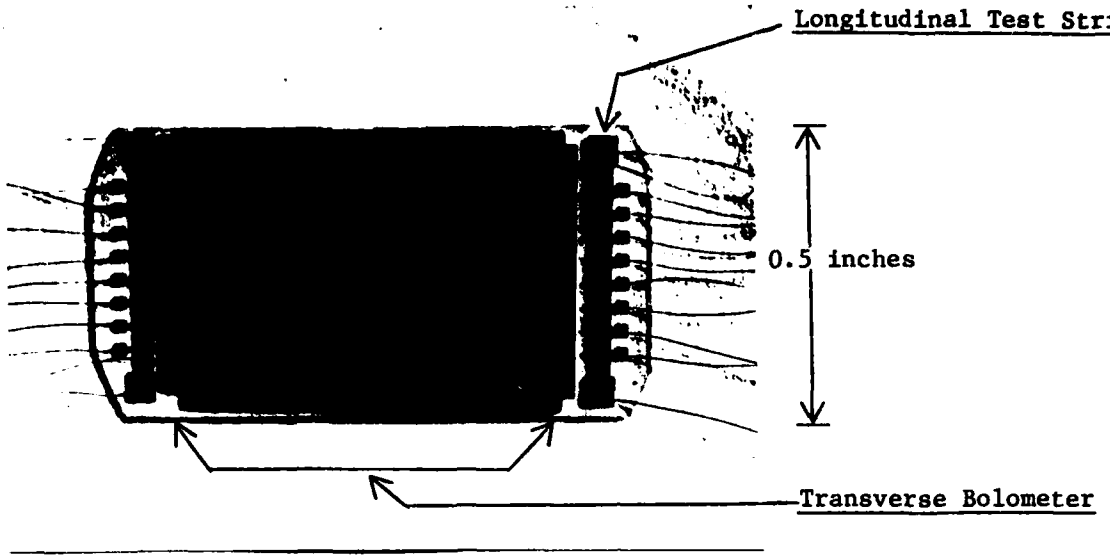


(b)

Fig. 2. (a) An array of longitudinal, four lead, bridge-configurated gold germanium resistors. Each resistor is about 0.08 inch x .04 inch x 2500 Angstroms thick. The sapphire was precut on the back side before evaporation. (b) An optical micrograph showing the active Ge/Au alloy film over the contact areas. The misalignment was due to the extreme angle of evaporation adopted to ensure good step coverage on the bolometers.



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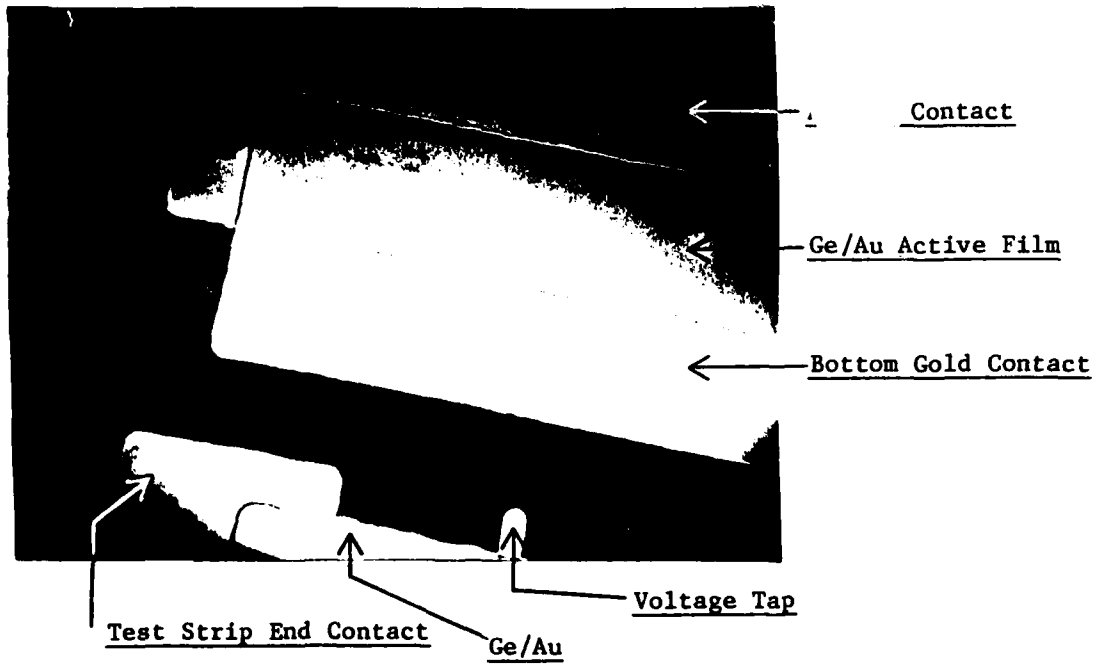


(b)

Fig. 3 (a) Bolometer film on a sapphire substrate contacted with superconducting wires. Test strips are at either end of the bolometer. (b) Enlargement of one corner of the bolometer showing the three layers and a part of test strip used for contact resistance and heat treatment monitoring.

The chromium bottom adhesive layer was evaporated from a Cr plated filament, followed by the bottom gold contact, about 1500A thick, the Au/Ge active layer (500A to 3500A thick, flash evaporated) and the top gold contact. The gold was evaporated from tungsten dimple boats. By waiting about thirty minutes between evaporations, the substrate temperature rise could be kept to only a few degrees Celsius.

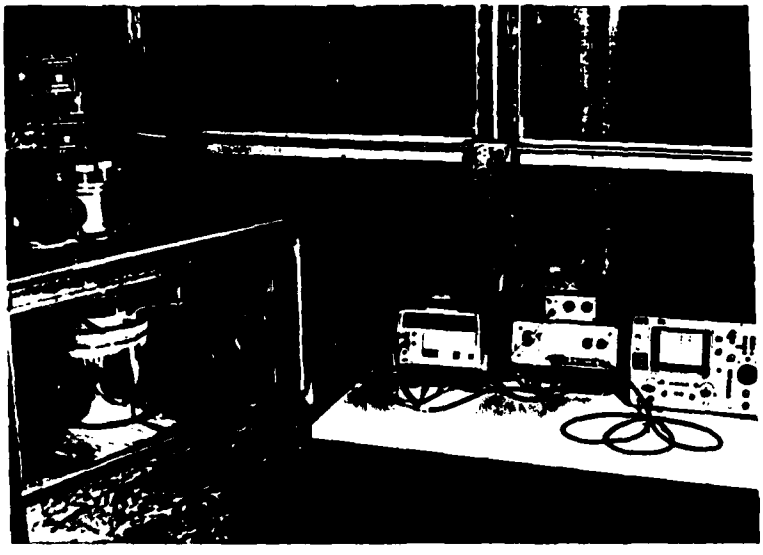
The resistance/temperature characteristics were measured by a SQUID picovoltmeter [2] in a cryostat that could be varied between 4.2K and 1.5K by reducing the pressure over the liquid helium. The set-up is shown in Fig. 4. No changes had to be made in the SQUID electronics for these measurements. The high resistance longitudinal measurements were made on the Lake Shore calibration system with a current source and high impedance voltmeter capable of one microvolt resolution.

The superconducting leads, chosen because the noise figure of the bolometer depends strongly on the heat leak to the outside world, were made by evaporating Indium onto Kevlar [3] filaments and then cold welding them to the contact pads with Indium. Great care must be exercised in doing this, but no insurmountable problems were encountered.

Results

The Au/Ge films behaved as expected from previous work in the high resistance longitudinal mode. The as-evaporated films had an approximately power-law dependence on temperature, as the example in Fig. 5 shows. Upon heat treating at about 200°C for a few minutes, the resistors showed increased sensitivity. After treatment at 350°C (near the Au-Ge eutectic temperature) the sensitivity and resistance decreased, as shown in Fig. 6. The as-evaporated films are reported to be amorphous [4], although that was not confirmed independently. It is expected that after heat treatment at elevated temperatures, various degrees of crystallization are achieved.

Potentially useful differences between heat treated and unheat-treated films are also seen in the magnetoresistance curves of Figs. 7 and 8 [5].



Cryostat and
SQUID Probe

Measuring
Instruments

Fig. 4. Experimental apparatus for measuring bolometer resistance with the SQUID picovoltmeter.

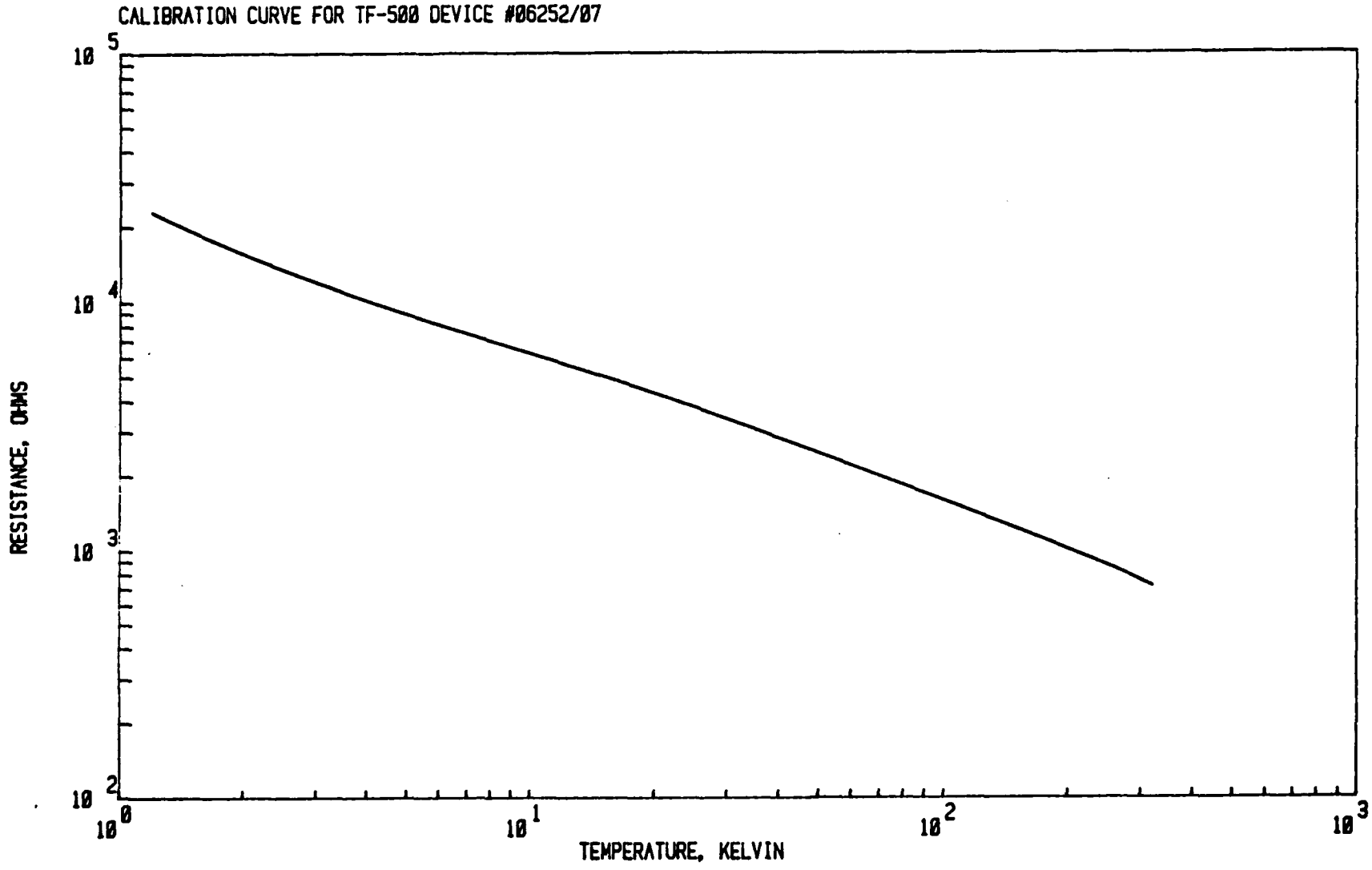


Fig. 5. Temperature/resistance characteristic for an un-heat treated 16% Au/Ge film in the longitudinal mode.

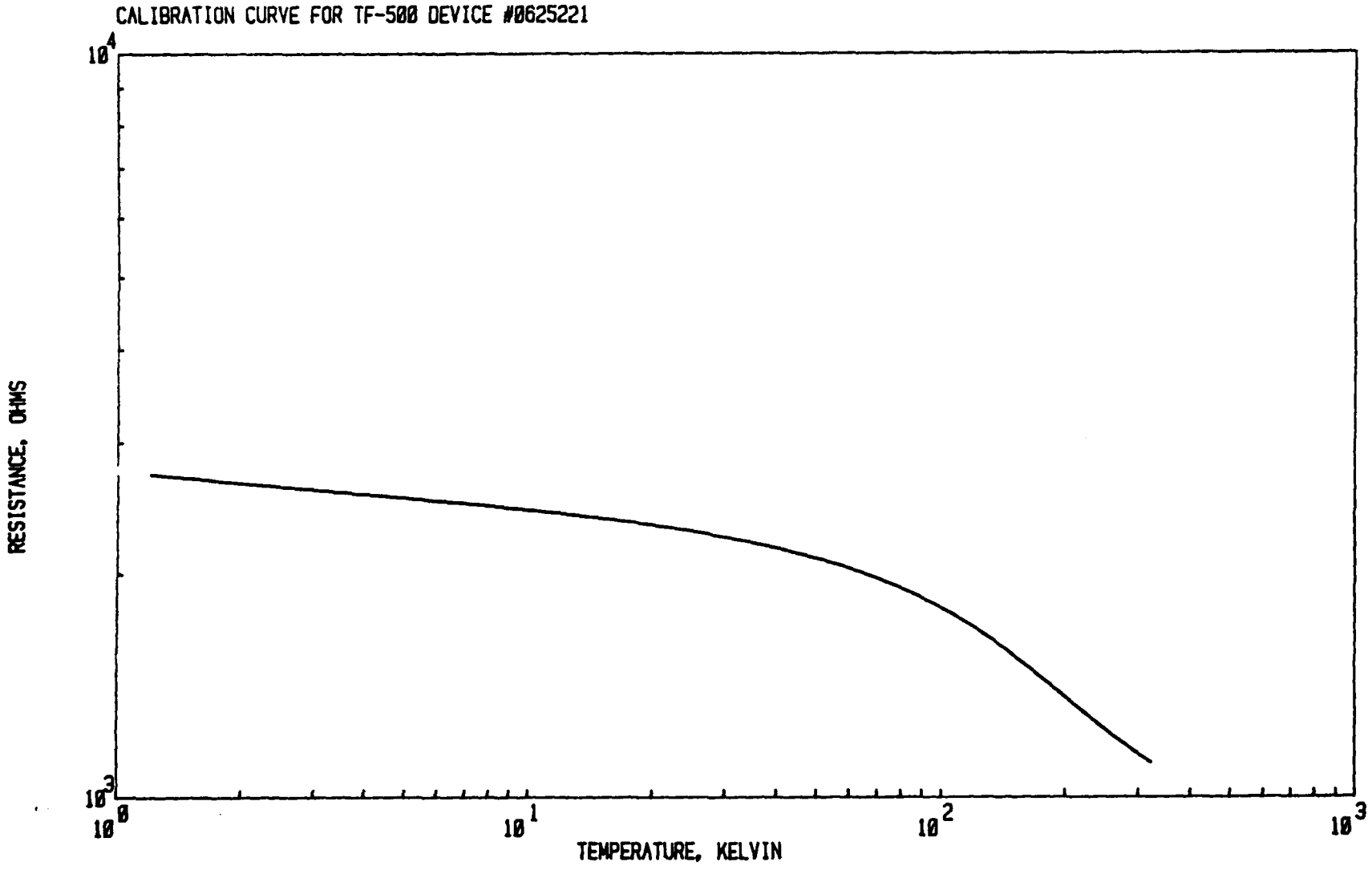


Fig. 6. Temperature/resistance characteristic for a 16% Au/Ge film resistor heat treated at 350°C for five minutes in forming gas.

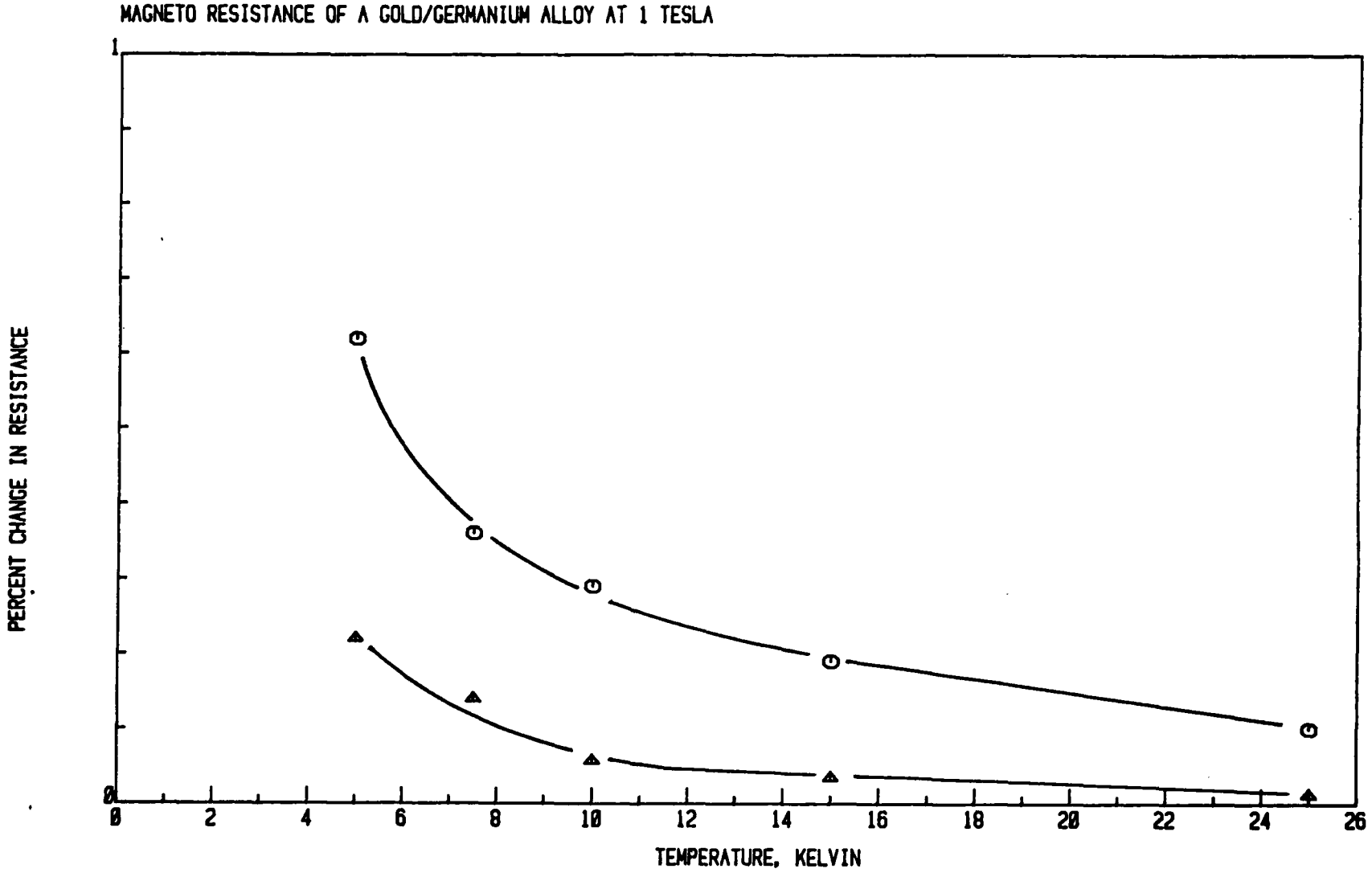


Fig. 7. Magnetoresistance of a 16% Au/Ge alloy film resistor over a limited temperature range. Film thickness is 2500A. Note that at this field strength the magnetic effect on the un-heat treated film is greater than that on the heat treated film. △ Sample 06252/007. ○ Sample 06252/021.

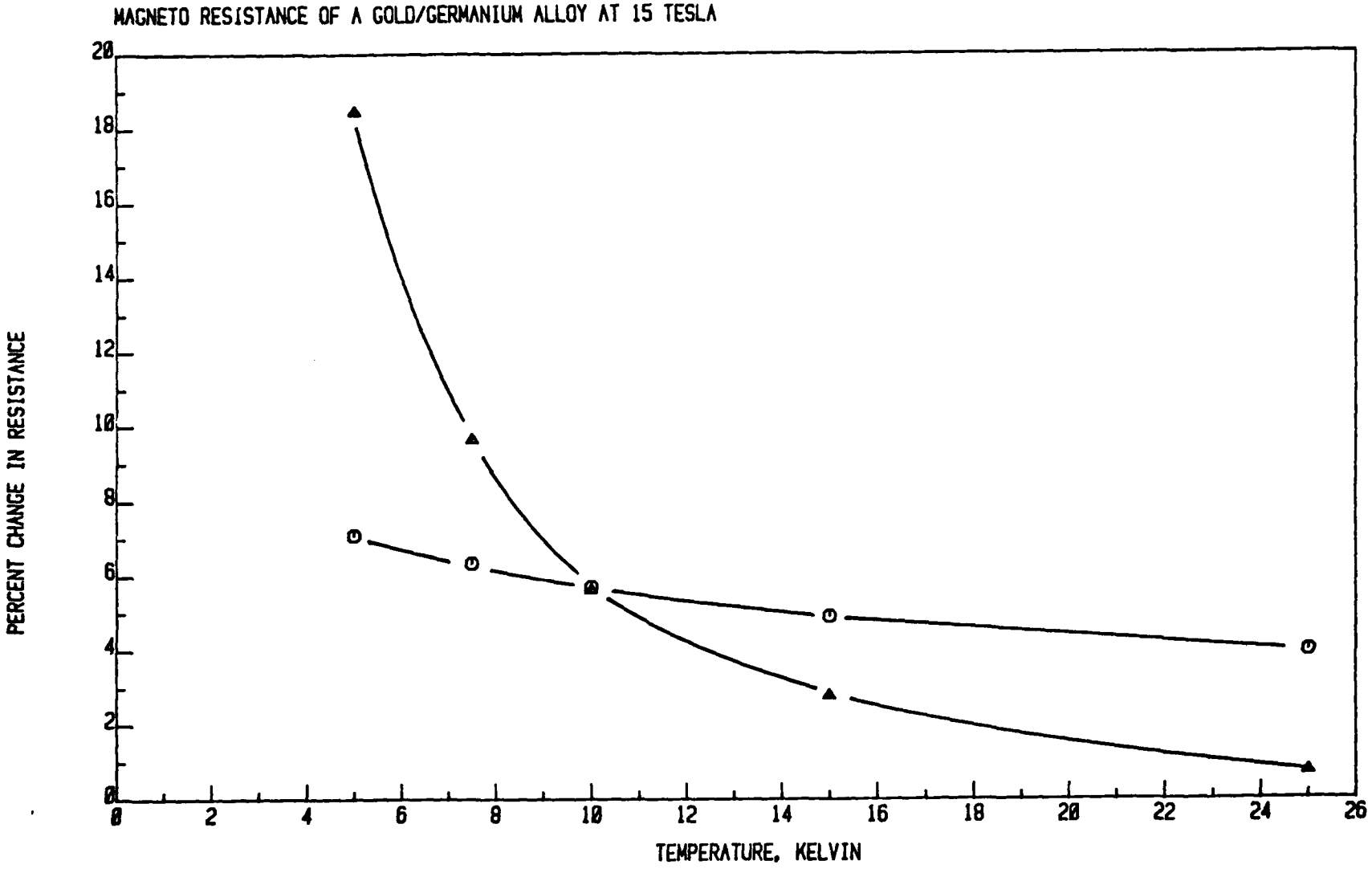


Fig. 8. Magnetoresistance of a 16% Au/Ge alloy film resistor over a limited temperature range. Film thickness is 2500A. Note that the curves have crossed at 10K at the higher field strengths (refer to Fig. 7). △ Sample 0625/007. ⏲ Sample 06252/021.

The effects of magnetic fields are in general quite low compared to that of doped bulk germanium (a factor of 10 at 15 Tesla), but specifically, the heat treated films stay quite low in magnetoresistance while the un-heat treated films continue to show increased effects.

The contact resistance also decreases upon heat-treatment, as shown in Fig. 9. When plotted on a greatly expanded scale, this data yields a contact resistance [1] of 0.135 ohms for the unheated films at 300K, but is not measurable to any degree of accuracy for the heated films, indicating a significant improvement.

It was surprising to find that the transverse resistance of the bolometers had the opposite resistance characteristic from the longitudinal resistance. Fig. 10 shows that it was in fact characteristic of metals. These same results were obtained for thicknesses from 500A to 3500 A. The best data obtained to explain this phenomena was supplied by Mochel [4] from transmission electron micrographs of 500A films. They reportedly show that the gold particles vary in size from 20A to almost the thickness of the film, and hence could "short out" the film in the transverse direction.

In an attempt to reduce the gold particle size, a thermoelectric module was placed on top of the sample holder, and a 2kg aluminum heat sink was placed on top of the module. After the bottom contact was formed, the sample was cooled to -60°C and the active Au/Ge film was flash evaporated. The sample was warmed and the top Au contact was made. The Al heat sink overheated, however, and the sample reached about 40°C at one point before it was removed from the holder. This may have caused some grain growth. The net result was an increase in the bolometer resistance and a decrease in the ratio of 300K resistance to 4.2K resistance. Both effects are in the expected direction for decreased gold particle size, although the evidence is hardly exhaustive or conclusive.

Extensive materials analyses were done at an independent surface analysis laboratory. The results are presented in the appendix. SEM examination did not reveal the Au particle sizes that the TEM studies did at the University of

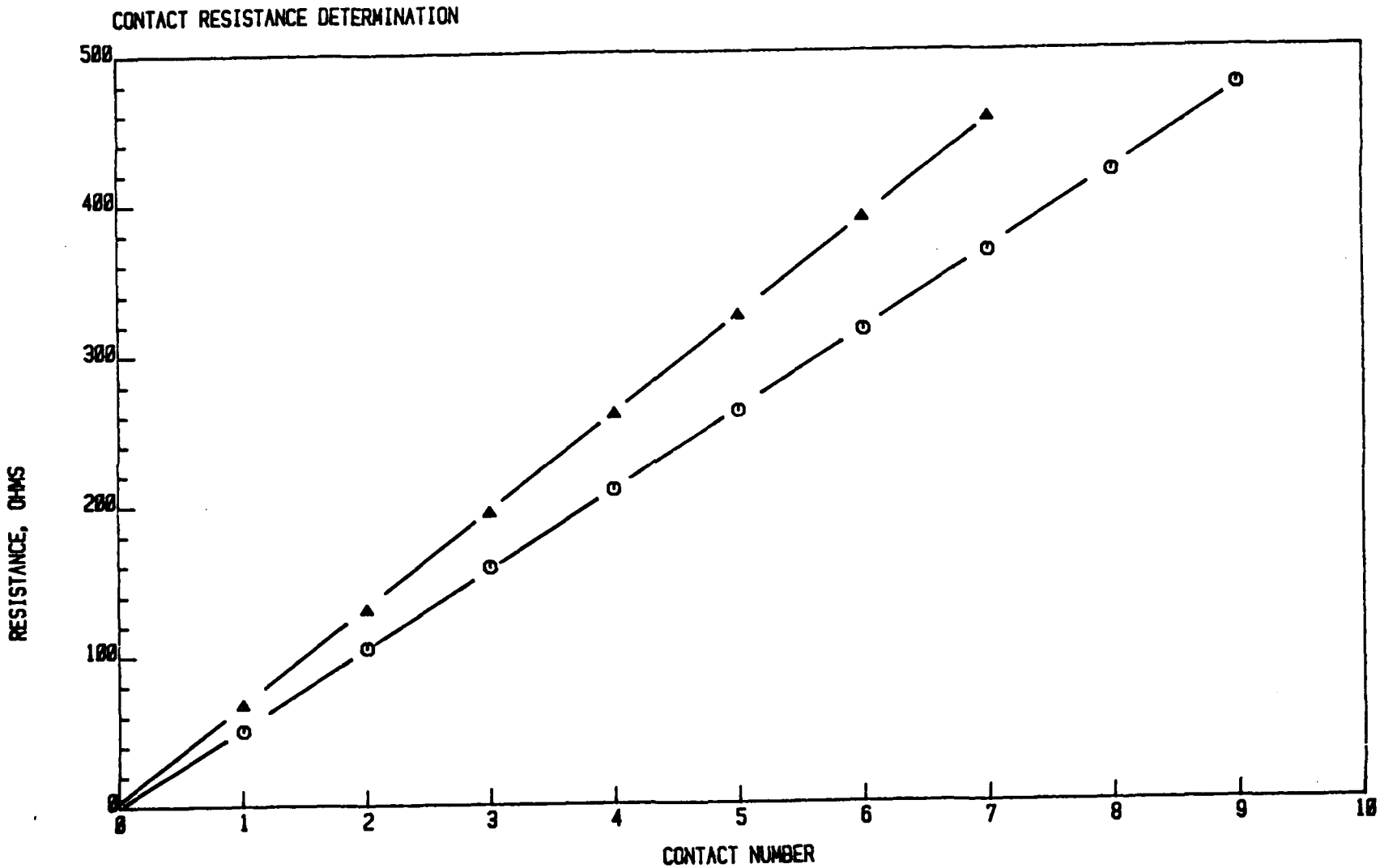


Fig. 9. Contact resistance determination by Schockley's method from a test strip on a bolometer substrate. Film is 16 atomic %Au in Ge. Thickness is 2300A.

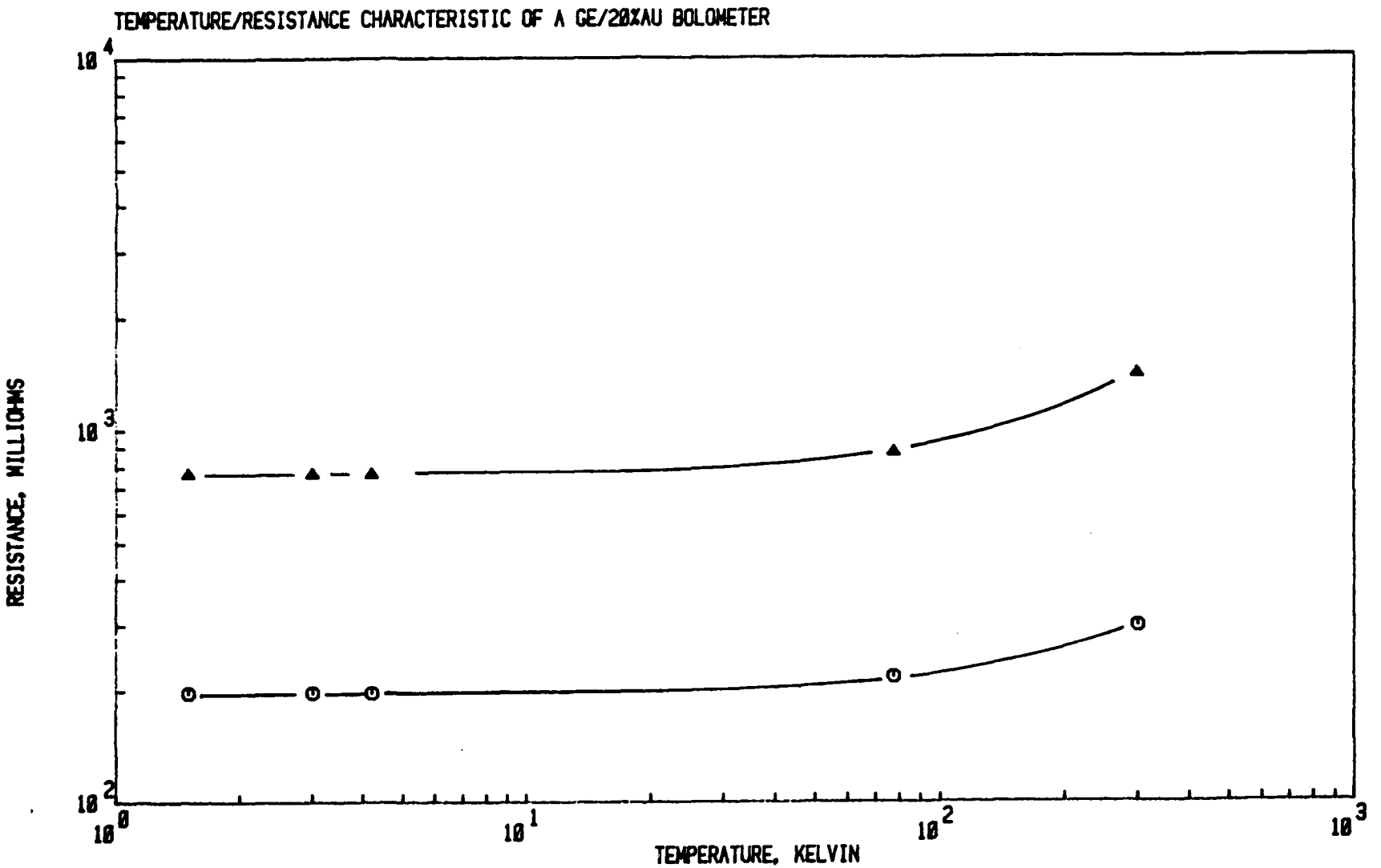


Fig. 10. Temperature/resistance characteristic of 20% Au/Ge bolometers deposited at 300K (○) and 213K (△) substrate temperatures.

Illinois. They did observe extensive phase separation in the films heat treated at greater than 300°C, however, which corroborates the results of the electrical measurements, i.e., reduced magnetic field effects, sensitivity and contact resistance. Many impurities were found, but since the high gold content would be expected to overwhelm any other metal ion content, and no effort could keep the samples clean during testing, the presence of these impurities is not considered significant.

Recommendations

Even though the "shorting out" of the Au/Ge films was a surprise, and a solution was not readily available, there are many reasons to continue with this line of inquiry. It is still very desirable to have a bolometer-type detector that could be matched to a SQUID amplifier, for reasons of ease of use, low noise, high gain and high dynamic range. The actual fabrication and testing of these devices was relatively simple, so the following recommendations are made:

1. Continue with the gold/germanium size films to try to control the gold particle size and distribution:
   a. Co-evaporate the gold and germanium from separate boats.
   b. Sputter the films.
   c. Perform (a.) above on cooled substrates to prevent the gold from coalescing, perhaps as low as 77K.
2. Search for either a theoretical reason why the transverse resistance of a metal/semiconductor alloy should be metallic, or a "metal/semiconductor" combination that might be more controllable, such as Pt/Si, Au/C, or C/SiC.
3. Investigate other types of materials such as the charge density wave material NiSe:Fe [6], which has an extremely low resistivity above approximately 50K, but then shoots up with a huge d(lnR)/d(lnT) of about 8 at 4.2K.
4. Expand the concept of a bolometer/SQUID system to DC SQUIDs which can be impedance matched at 10 ohms. This may allow a much wider range of materials to be used, in either the transverse or longitudinal modes.
   The operation of the DC SQUID in a non-flux-locked mode, contrasted to the rf-biased SQUIDs, may enhance noise immunity and other signal processing aspects also.

5. Limit investigations to the range of 4.2K, so that practical systems using mechanical refrigerators or cryostats may be constructed.

6. Define the desired performance criteria for a bolometer/SQUID detector.

7. Begin design investigations into possible SQUID/bolometer hybrid or monolithic structures that would incorporate the necessary thermal and electrical features to reach the goals of (6).

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2. S.H.E. Corporation.

3. Kevlar and procedure provided by Professor Paul Richards.

4. Mochel, J., University of Illinois Department of Physics, private communication.

5. Data taken at the Francis Bitter National Magnet Laboratory, Massachusetts Institute of Technology; trip funded independently by Lake Shore Cryotronics, Inc.

6. Hillenius, J., Coleman, R.V., Fleming, F.M., and Cava, R.J., Phy. Rev. B23, 1567 (1981).

Appendix A

Included are excerpts from surface analysis studies of gold-germanium alloy films of about 16 atomic per cent gold.

INTRODUCTION

Eight sets of thin film resistors made of a germanium/gold alloy were submitted to Scanning Electron Analysis Laboratories, Inc., for metallurgical evaluation. Most of the samples were supplied in duplicates (A and B). Sample groups 1-4 and 5-8 were made in different ways. The resistors were made by depositing 500 Å thick gold over chromium at the contact pad fingers on a sapphire substrate and depositing a 1000 Å thick Ge-Au (40 a/oAu) film over the contact pads and in the active area of the resistor. The following descriptions were provided for the samples submitted:

Sample Number	Description	Sample Number	Description
1A	Unheated 06252	1B	Unheated 06252
2A	160°C 06252	2B	160°C 06252
3A	350°C 06252	3B	350°C 06252
4A	Light Etch 06252	4B	Heavy 06252
5A	Unheated 06182	5B	Unheated 06182
6A	160°C 06182	6B	160°C 06182
7A	350°C 06182	7B	350°C 06182
8	Light Etch 06182		

RESULTS

A schematic diagram of the Ge/Au thin film resistor is shown in Figure 1. Typical optical photomicrographs of Sample Nos. 1A and 5A are shown in Figure 2.

Sample Nos. 1A through 7A and No. 8 were examined in a scanning electron microscope (SEM) to determine the surface texture and topography. The samples were coated with a thin film of carbon

using a vapor deposition technique to render electrical conductivity to the exposed sapphire surfaces and to make the samples suitable for SEM examination. The contact finger, contact pad and the active film areas, referred to as Areas A, B and C in Figure 2(a), respectively, were examined for all the samples and the SEM micrographs are presented in Figures 3 through 23. The films were thin and the film surface in the contact finger and the active film areas revealed replication of scratches on the polished sapphire substrate. The active films in Sample Nos. 5-8 were thicker than those of Sample Nos. 1-4. In general, the resistor surfaces were quite dirty, perhaps due to handling and atmospheric exposure after the thin films were deposited. No differences were observed between the surface textures of the unheated samples, samples exposed at 160°C and samples with a light etch. Exposure at 350°C, however, changed the surface texture of the contact pad area and the active film area in Sample No. 3A (thin active film) and of the contact pad area in Sample No. 7A (thicker active film), Figures 10, 11 and 21. The thin film (Sample No. 3A) appeared to recrystallize with a very fine grain size, however, the thicker film (Sample No. 7A) revealed larger topographic bumps, Figure 21(b). The light etch sample (No. 8) revealed etch pits in the Ge/Au layer and no structure was revealed, Figure 23.

The contact finger, contact pad and the active film areas, referred to as Areas A, B and C in Figure 2(a), respectively, were analyzed for local chemistry using an energy dispersive x-ray (EDX) microprobe analyzer. The EDX spectra obtained for Sample Nos. 1A through 7A and No. 8 are presented in Figures 24 through 47. It should be noted that the chemical information obtained by the EDX technique at a 20 kV beam voltage is from a depth of approximately 1 micron. Therefore, for thinner films, Al was picked up from the sapphire (Al_2O_3) substrate. Since the Ge/Au active film was thinner for Sample Nos. 1-4 as compared to Sample Nos. 5-8, a higher Al

peak from the sapphire was obtained for Sample Nos. 1-4. The Au over Cr contact fingers appear to have a similar thickness on both sets of samples. Both Sample Nos. 3A and 7A (baked at 350°C) revealed a higher gold peak than germanium in the contact pad area, indicating an outward diffusion of gold during the baking process. The chemistries obtained for the unheated sample, the sample baked at 160°C and with a light etch revealed a similar EDX spectra at identical locations for both sets of samples, individually.

The two phase structure observed on Sample No. 3A (baked at 350°C) revealed a light and a dark phase, identified as Areas A and B, respectively, in Figure 11(a). The EDX spectra revealed that the light phase was gold rich and the dark phase was germanium rich in two phase Ge/Au alloy, Figures 48 and 49.

Sample Nos. 1B through 7B were mounted in cross section and metallographically ground and polished. However, due to the thickness of the films and the hardness of sapphire, the polishing was extremely difficult and no meaninfgul data could be obtained.

Sample Nos. 1A and 5A (the unheated samples from both lots) were also subjected to an Ion Microprobe Mass Analysis (IMMA) using an $^{18}O_2^+$ ion beam and the positive secondary ions were analyzed by the Secondary Ion Mass Spectroscopy (SIMS) technique. The positive SIMS spectra obtained at Areas A, B and C of Figure 3 (Sample 1A) are presented in Figures 50, 51 and 52, respectively. The IMMA spectra were obtained after sputtering the surfaces in the bulk film. The films revealed heavy contaminations of C, Na, Al, Si, K, Ca, hydrocarbons, Ti and Co. It should be noted that the ionization yields for all the elements are different and the pertinent elements in the decreasing order of ionization yields are Li, Na, K, Ca, Al, Cr, Si, Ti, Co, C, Au and Ge. A depth profile for C, Na, Al, Si, K, Ca, Cr, Ge and Au was also obtained in the contact pad area (Area B, Figure 3) of Sample No. 1A and is presented in Figure 53.

Due to a complex composition of the film and different sputtering rates of different elements, the film layer thicknesses could not be determined. The surface shows a carbon film (which was vapor deposited to make the surface electrically conductive); a Ge/Au film with contaminations of Al, Na, K, Ca, Si and Cr; a gold rich film with Ge, Cr, Ca and Si contamination; a chromium film with a small amount of contaminants; followed by the sapphire (Al_2O_3) substrate, in that sequence, Figure 53.

An IMMA spectra for positive secondary ions obtained in the contact pad area of Sample No. 5A is presented in Figure 54. The Ge/Au bulk film revealed a very heavy contamination of Al, Na, Ca, Li, C, Si and Mg; Al contamination being the highest. The overall contamination was much larger than that observed for Sample No. 1A. A depth profile was obtained for Li, C, Na, Al, Si, Ca, Cr, Ge and Au in the contact pad area of Sample No. 5A and is presented in Figure 55. Due to a high aluminum contamination, the sputtering rate was much slower and the depth profile was obtained for 2.75 hours to reach the sapphire substrate. The top surface contained a carbon film (deposited for SEM/IMMA analyses); a Ge/Au film with contamination of Al, Ca, Na, Si, Li and Cr; a gold film with Na, Li, Si, Cr and C contamination; a chromium film with a small amount of contamination; followed by the sapphire (Al_2O_3) substrate, Figure 55.

SUMMARY

SEM examination revealed dirty film surfaces, perhaps due to handling and atmospheric exposure after the thin films were deposited. The surface textures of the unheated samples, samples exposed at 160°C and samples with a light etch (for the thinner active film) were similar and revealed the replication of surface polishing scratches on the sapphire substrate. The active films in Sample Nos. 5-8 were thicker than those of Sample Nos. 1-4; the Au over Cr film in the contact finger

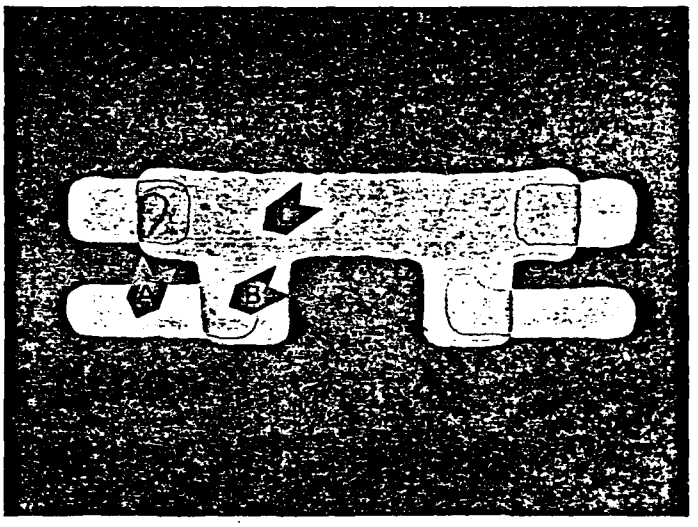
areas were of a similar thickness on both sets of samples. The thin film (Sample 3A) recrystallized in the contact pad area after a 350°C treatment, revealing a 0.25 micron grain size two-phase structure - a darker phase which was rich in germanium and a lighter phase which was rich in gold. The thicker film sample (Sample No. 7A) after being heated at 350°C revealed a wavy structure in the contact pad area. Light etching of the thick film sample (No. 8) revealed etch pits and did not reveal any microstructure.

The contact pad areas revealed a higher amount of gold after both sets of samples were baked at 350°C (Sample Nos. 3A and 7A), indicating an outward diffusion of gold from the Au over Cr film into the Ge/Au layer. Excess diffusion of Au into the Ge/Au may tend to locally change the composition to the Ge/Au eutectic composition (27 a/o Ge) with a melting point of 356°C. It should be noted that other contaminants in the Ge/Au film may further reduce the eutectic melting point.

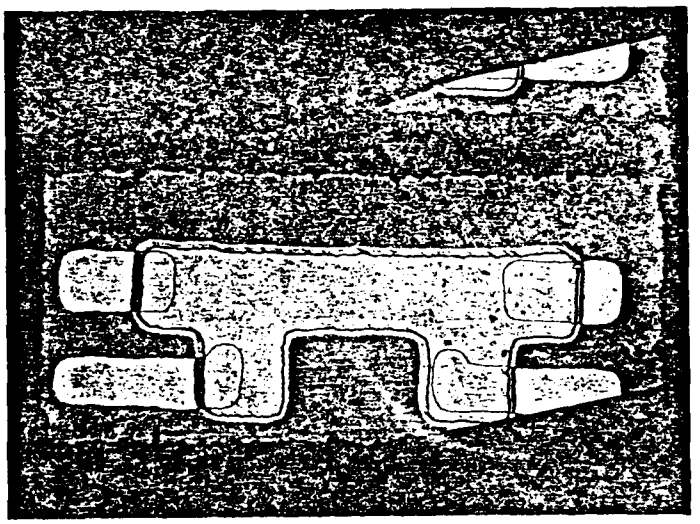
The IMMA results indicate a heavy contamination in the deposited films of Ge/Au as well as Au over Cr. Typical contaminants in the films were C, Li, Na, Al, Si, K, Ca, Ti, Cr, Co and hydrocarbons. The thicker Ge/Au film (Sample No. 5A) revealed a very heavy contamination of Al along with other contaminants. No tungsten contamination was found. In general, the film contamination may have been due to a lower vacuum, a dirty vacuum chamber with previous use of another type of film, and/or the use of lower purity starting materials for the thin film deposition. The surface of sapphire should be thoroughly cleaned prior to the deposition of thin films.

RECOMMENDATIONS FOR FURTHER WORK

Sample Nos. 1A, 2A, 3A, 5A, 6A and 7A should be analyzed further using the Ion Microprobe Mass Analyzer.

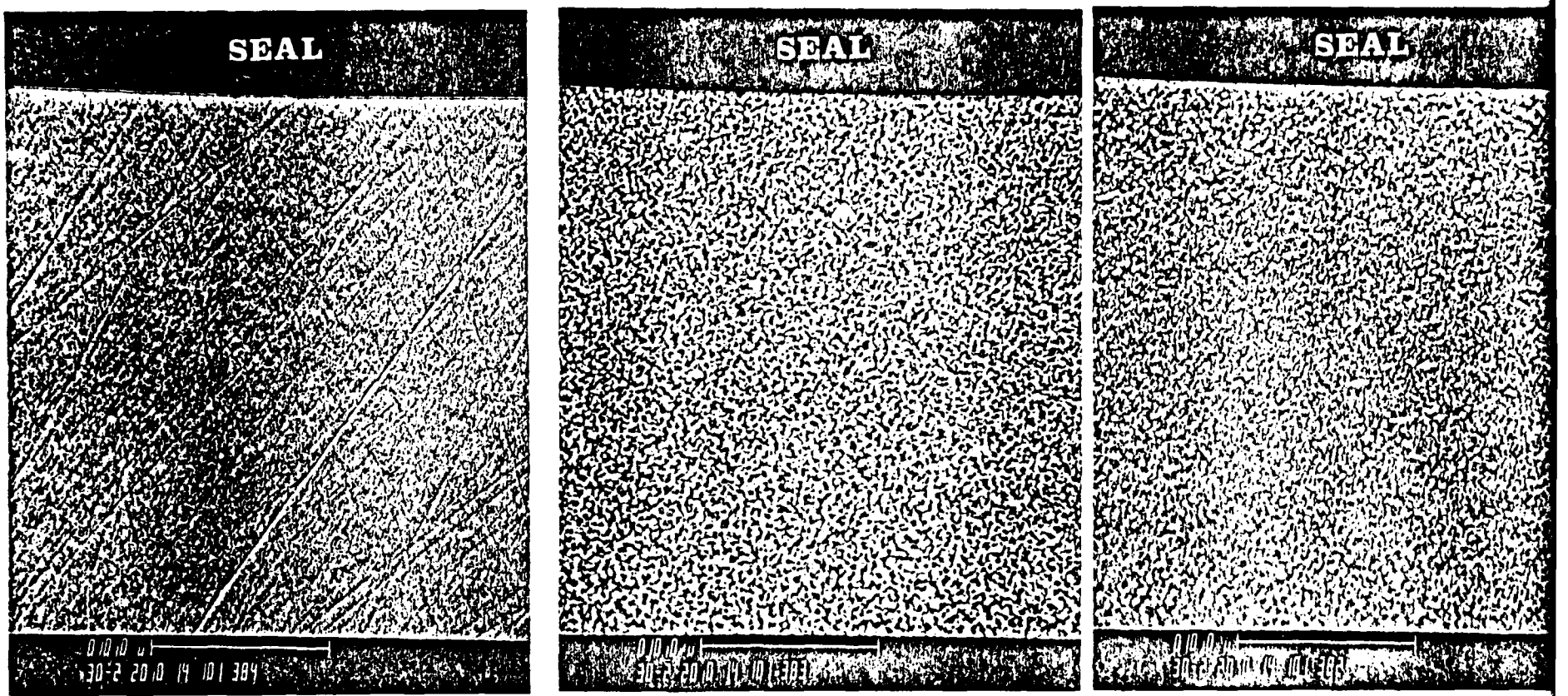


a.



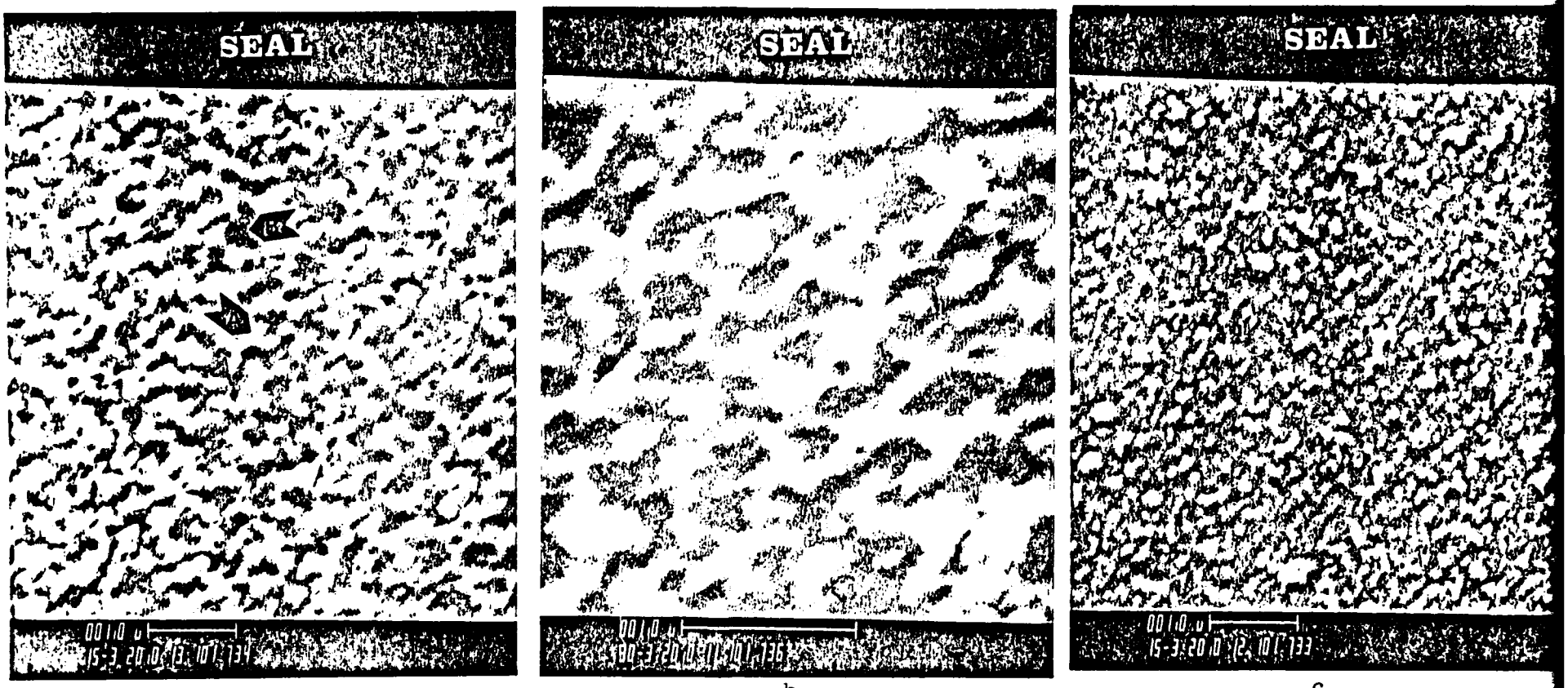
b.

Figure 2. (a) and (b) are the optical micrographs of resistor Sample Nos. 1A and 5A, respectively. (a) 31X (b) 31X



a. b. c.

Figure 10. (a), (b) and (c) are the detailed SEM micrographs obtained at Areas A, B and C of Figure 9, respectively. (b) and (c) reveal recrystallization of the Ge/Au film after heating at 350°C. (a) 101-384, 3000X (b) 101-383, 3000X (c) 101-382, 3000X



a. b. c.

Figure 11. (a), and (b) are the detailed SEM micrographs of Area B shown in Figures 9 and 10(b). The light areas are gold - rich and the dark areas are Ge-rich in the two phase structure. (c) is a detailed SEM micrograph of Area C shown in Figures 9 and 10(c). A large volume fraction of Ge-rich phase is observed in the active film as compared to the contact pad. (a) 101-734, 15,000X (b) 101-736, 3,000X (c) 101-733, 15000X.

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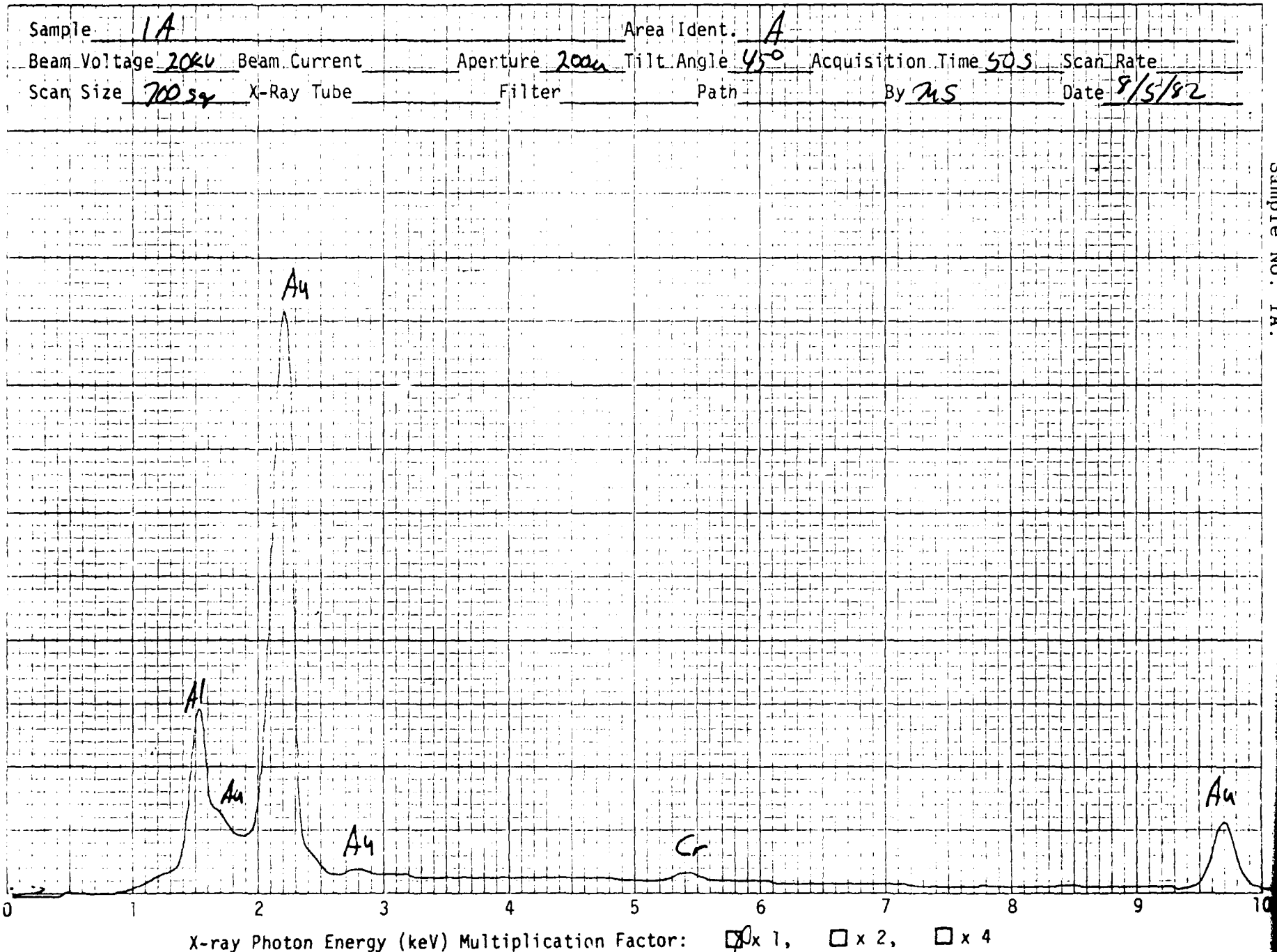
FLUORESCENT X-RAY SPECTRUM

Sample 1A Area Ident. A

Beam Voltage 20KV Beam Current ____ Aperture 200μ Tilt Angle 45° Acquisition Time 50S Scan Rate ____

Scan Size 700 sq X-Ray Tube ____ Filter ____ Path ____ By MS Date 8/5/82



Sample No. 1A.

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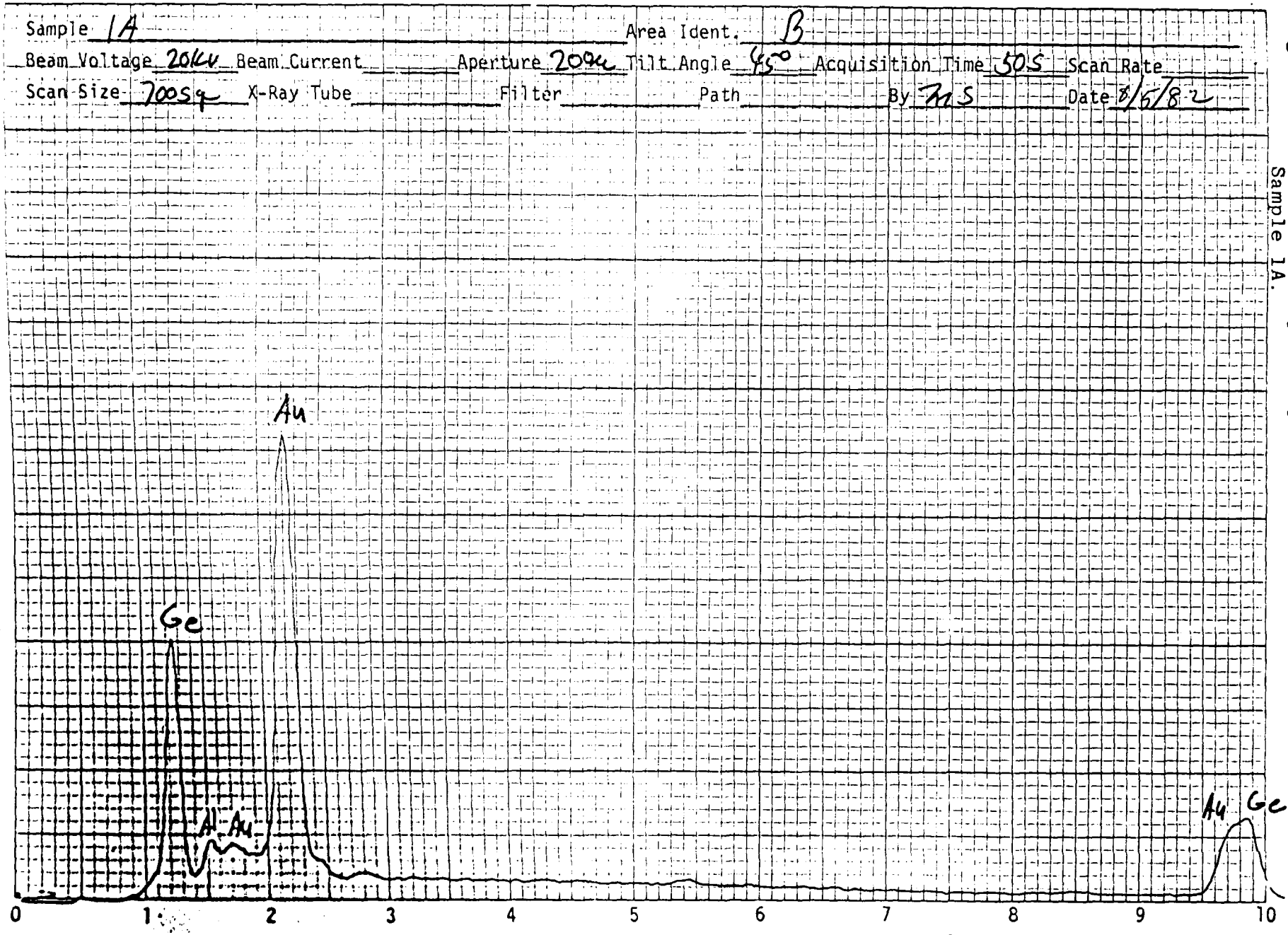
FLUORESCENT X-RAY SPECTRUM

Sample 1A Area Ident. B

Beam Voltage 20kv Beam Current ____ Aperture 20μ Tilt Angle 45° Acquisition Time 50S Scan Rate ____

Scan Size 700μ X-Ray Tube ____ Filter ____ Path ____ By MS Date 8/5/82



Sample 1A. Fluorescence spectrum obtained at Area B of

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FLUORESCENT X-RAY SPECTRUM

Sample 1A Area Ident. C
Beam Voltage 20KV Beam Current ____ Aperture 200u Tilt Angle 45° Acquisition Time 60 S Scan Rate ____
Scan-Size 700 SQ X-Ray Tube ____ Filter ____ Path ____ By RS Date 8/5/82

Total X-Ray Photon Counts (Full Scale 8192 Counts)

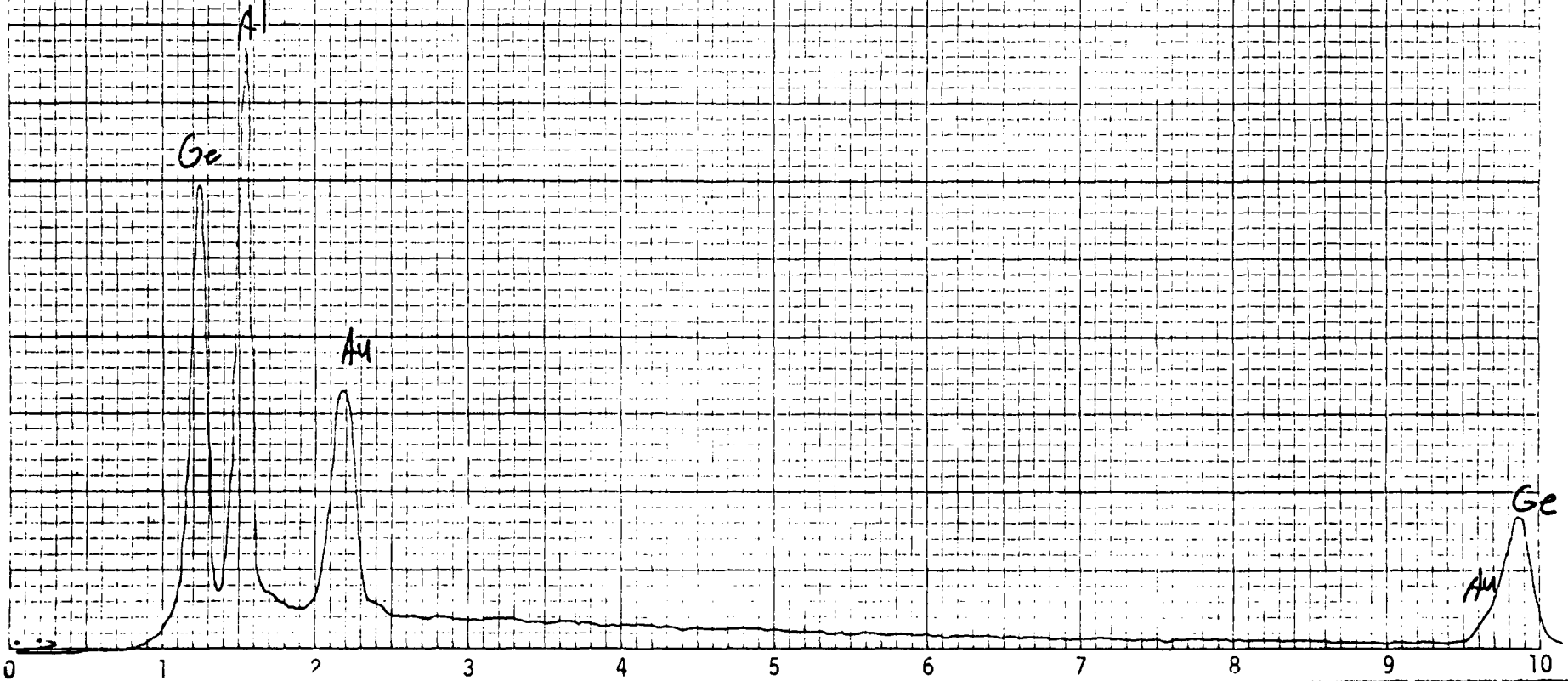

Figure 26. An EDX microprobe spectrum obtained at Area C of Sample No. 1A.

FLUORESCENT X-RAY SPECTRUM

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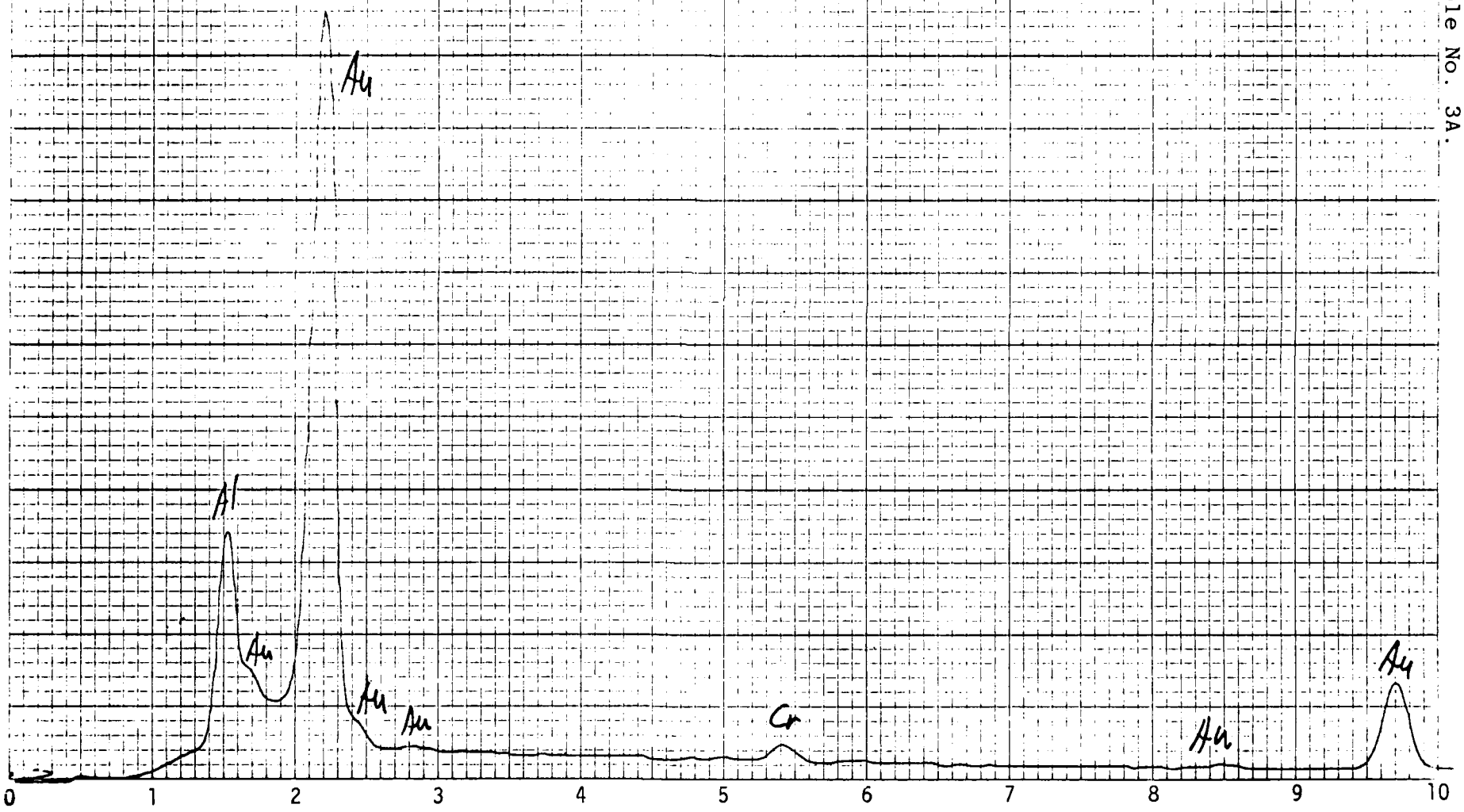
Sample 3A Area Ident. A

Beam Voltage 20KV Beam Current _____ Aperture 200μ Tilt Angle 45° Acquisition Time 50S Scan Rate _____

Scan Size 200sq X-Ray Tube _____ Filter _____ Path _____ By MS Date 8/5/82



Multiplication Factor: ☒ x 1, ☐ x 2, ☐ x 4

Figure 30. An EDX microprobe spectrum obtained at Area A of Sample No. 3A.

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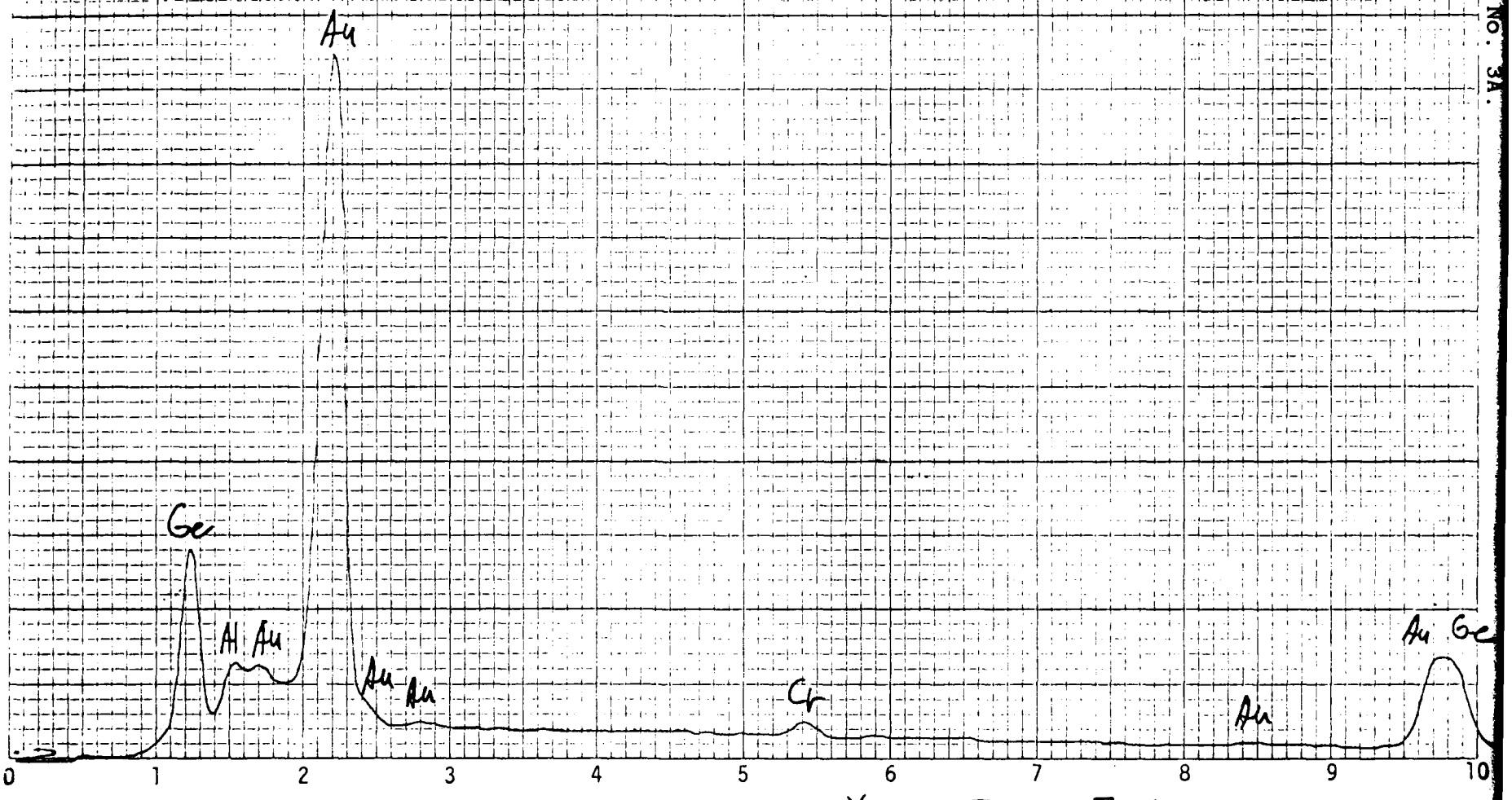
FLUORESCENT X-RAY SPECTRUM

Sample 3A Area Ident. B
Beam Voltage 20kV Beam Current ____ Aperture 200μ Tilt Angle 45° Acquisition Time 50s Scan Rate ____
Scan Size 700μ X-Ray Tube ____ Filter ____ Path ____ By MS Date 5/5/82

Total X-Ray Photon Counts (Full Scale 8/92 Counts)

Sample No. 3A



X-ray Photon Energy (keV) Multiplication Factor: ☒ x 1, ☐ x 2, ☐ x 4

FLUORESCENT X-RAY SPECTRUM

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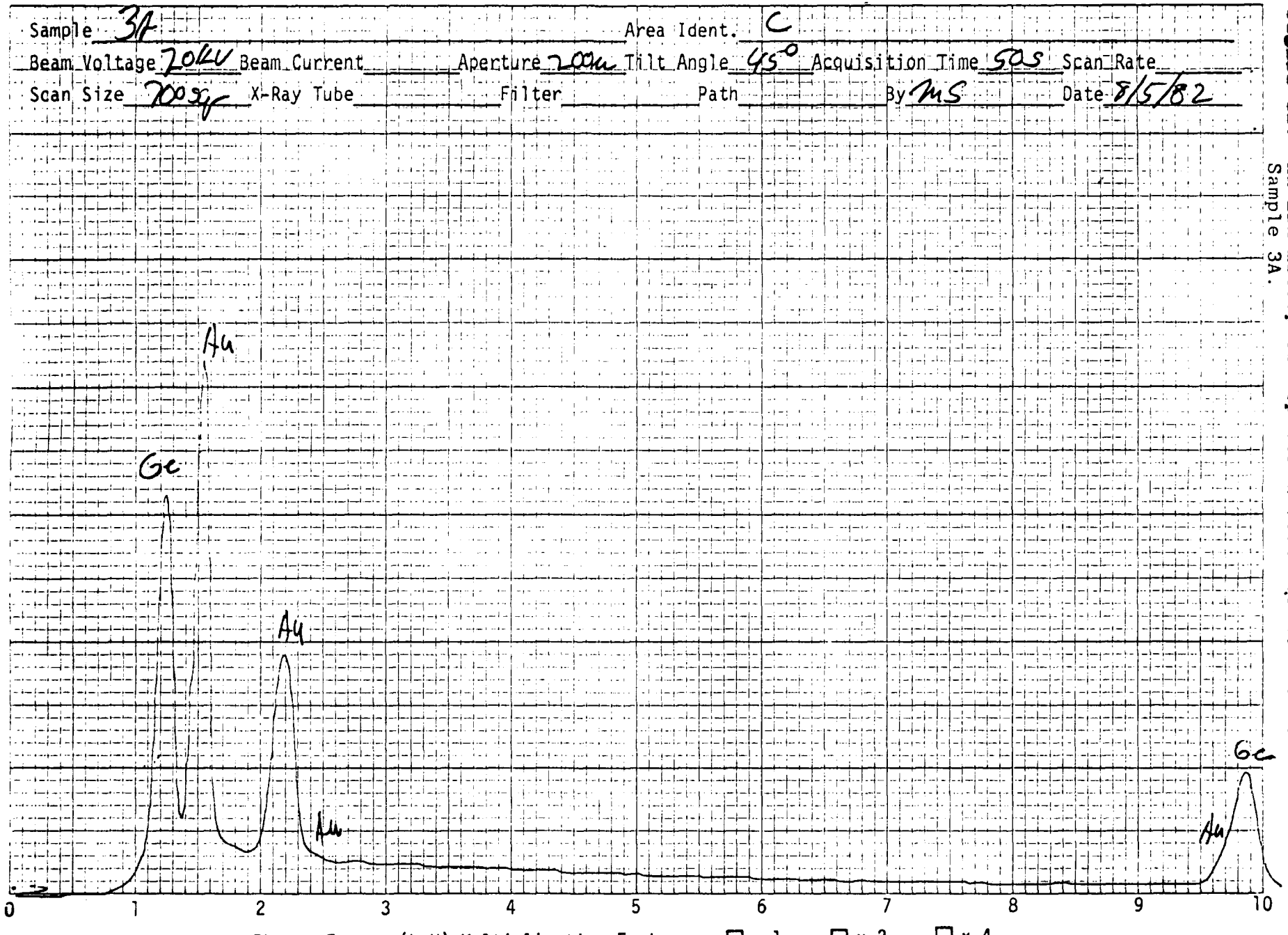
Sample 3A Area Ident. C

Beam Voltage 20kV Beam Current ____ Aperture 200μ Tilt Angle 45° Acquisition Time 50S Scan Rate ____

Scan Size 200μ X-Ray Tube ____ Filter ____ Path ____ By MS Date 8/5/82



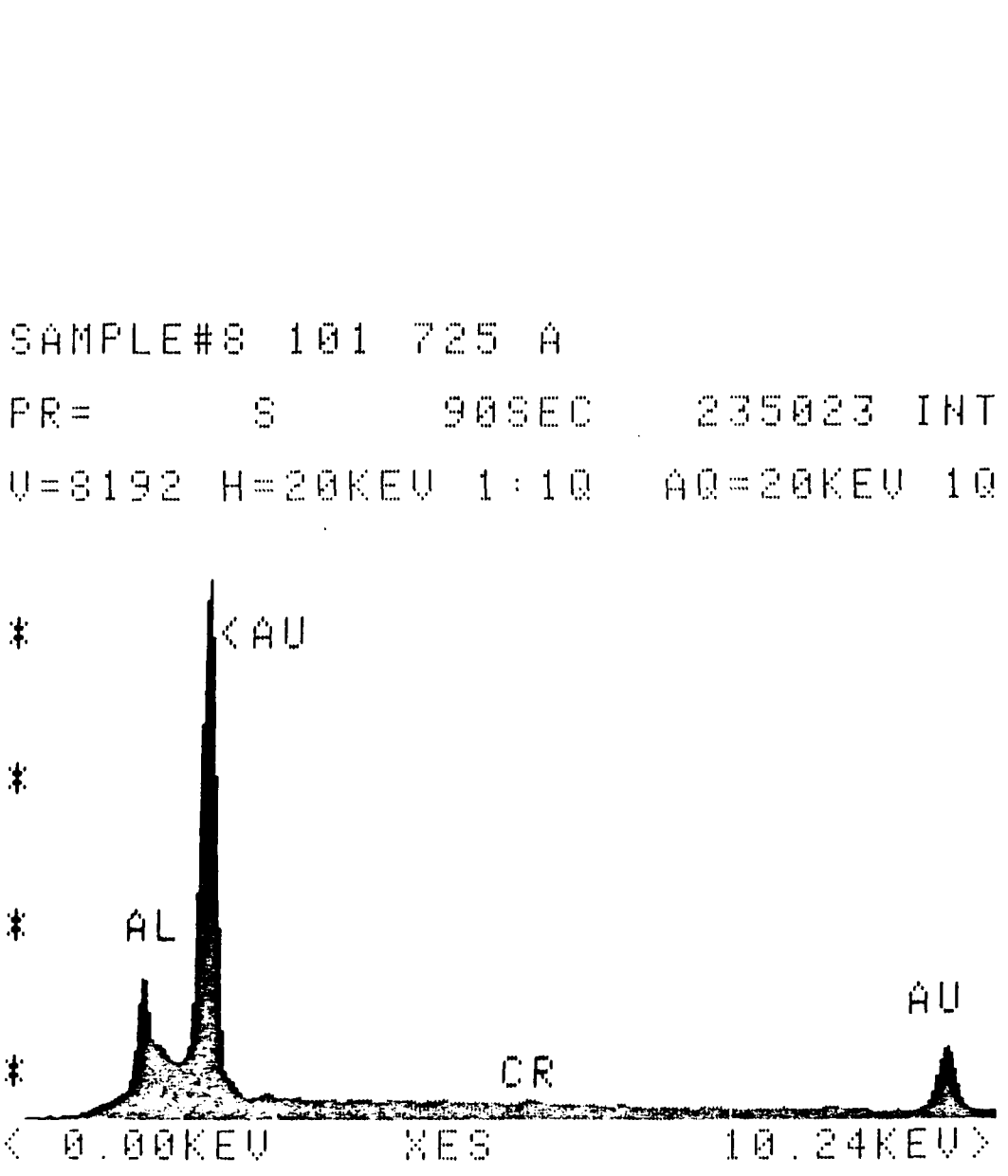


Figure 45. An EDX microprobe spectrum obtained at Area A of Sample No. 8.

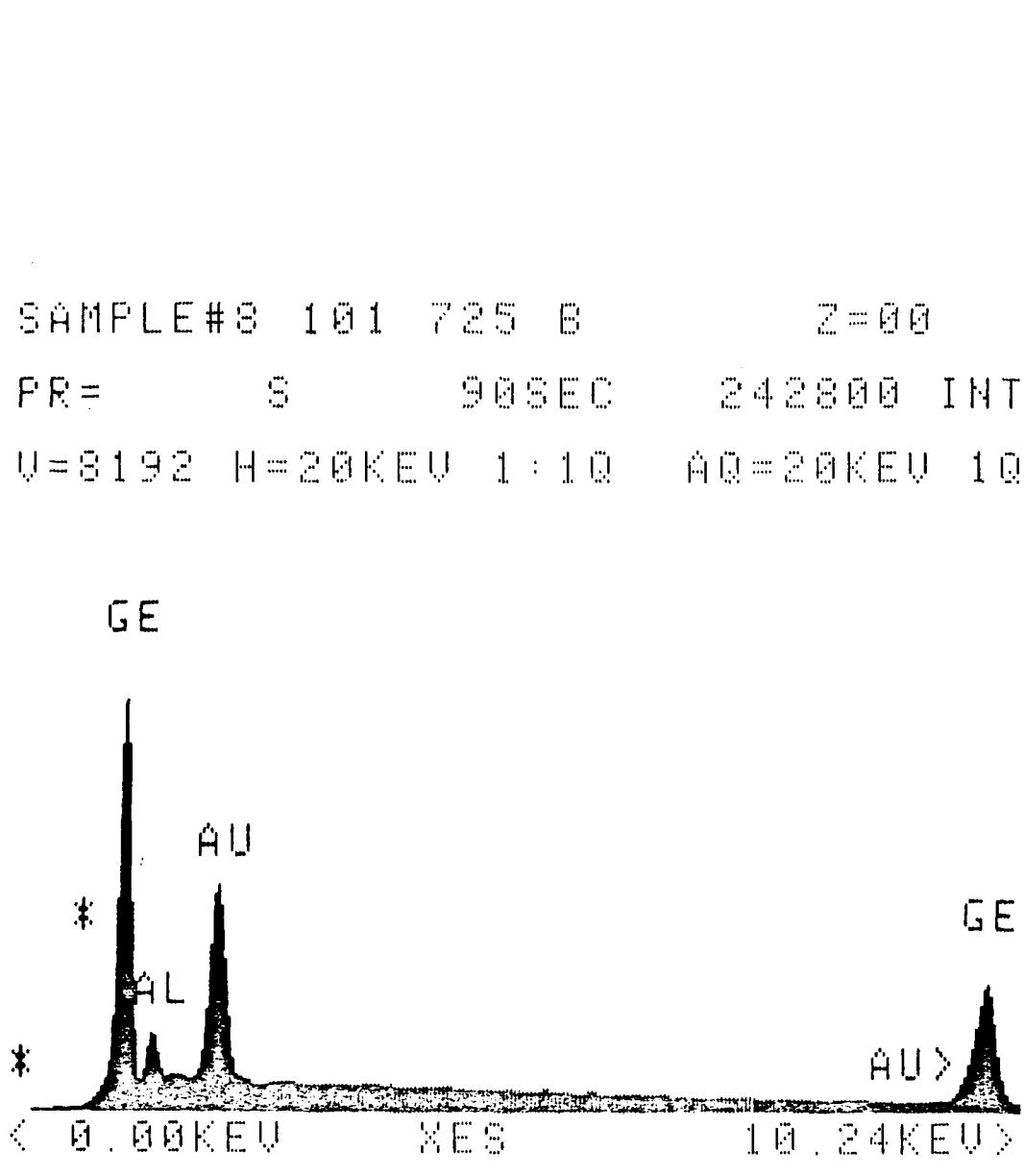


Figure 46. An EDX microprobe spectrum obtained at Area B of Sample No. 8.

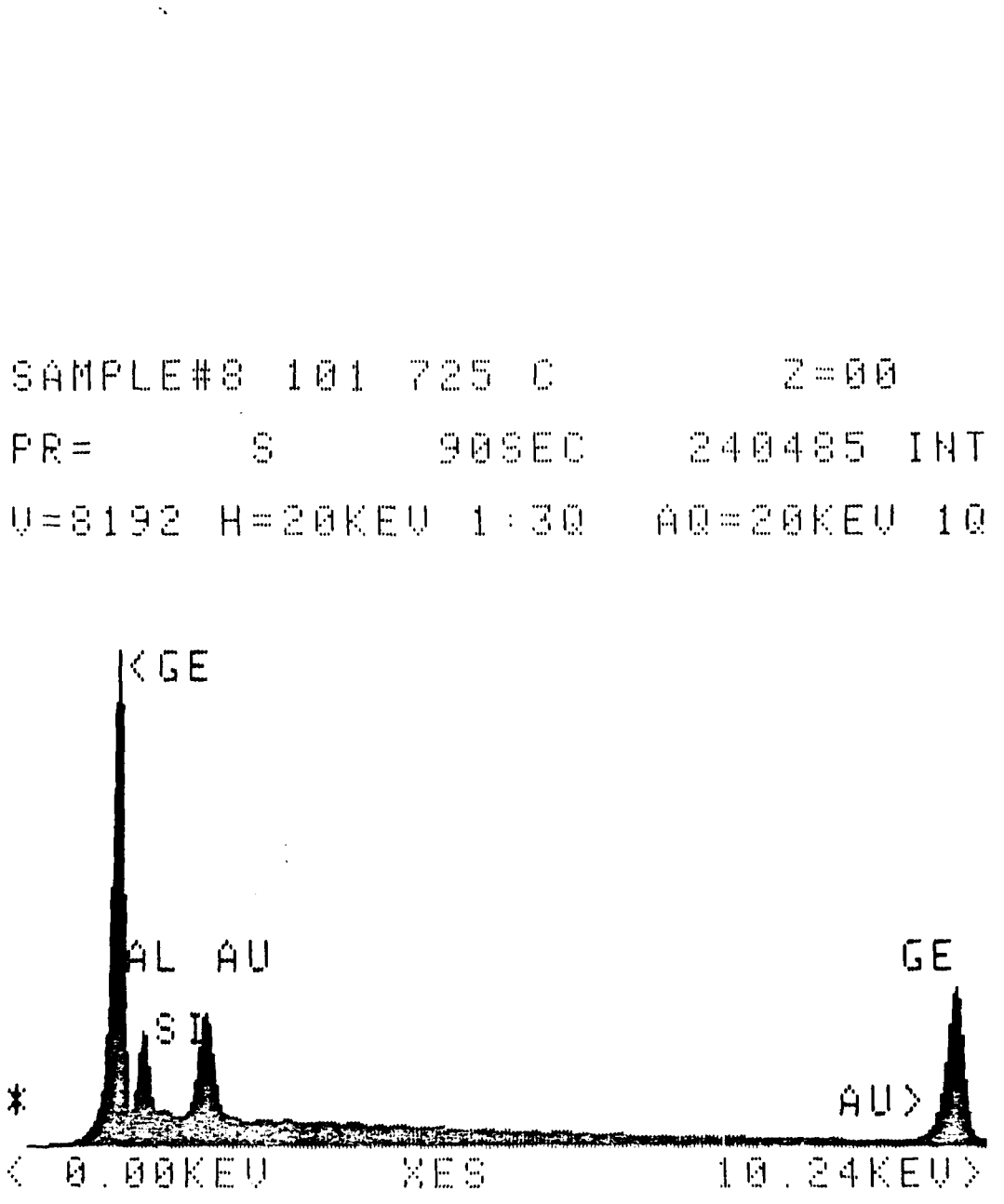
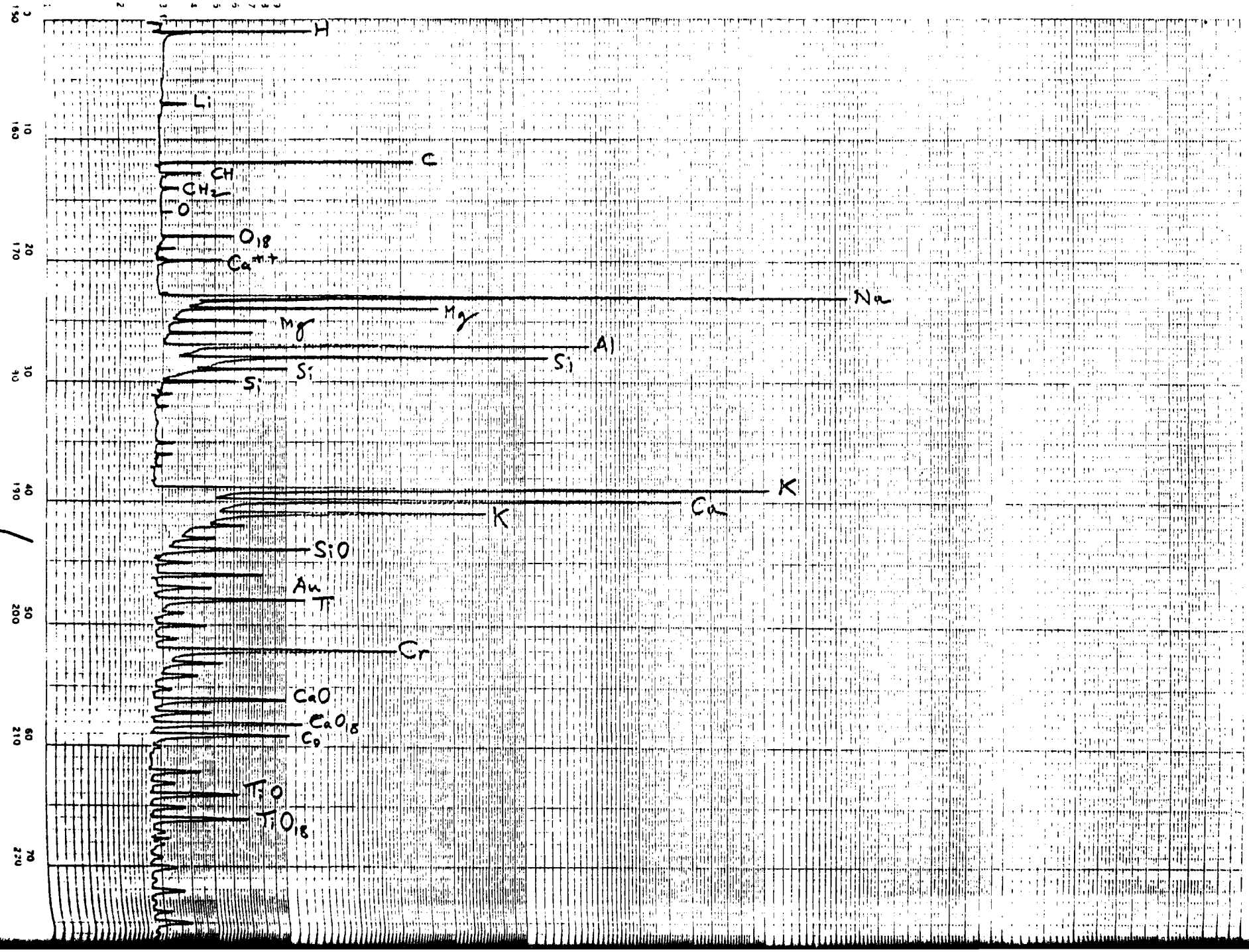


Figure 47. An EDX microprobe spectrum obtained at Area C of Sample No. 8.

AEROSPACE FORM 5134 K&E SEMI-LOGARITHMIC 5 CYCLES X 150 DIVISIONS KEUFFEL & ESSER CO. MADE IN U.S.A.

47 6210



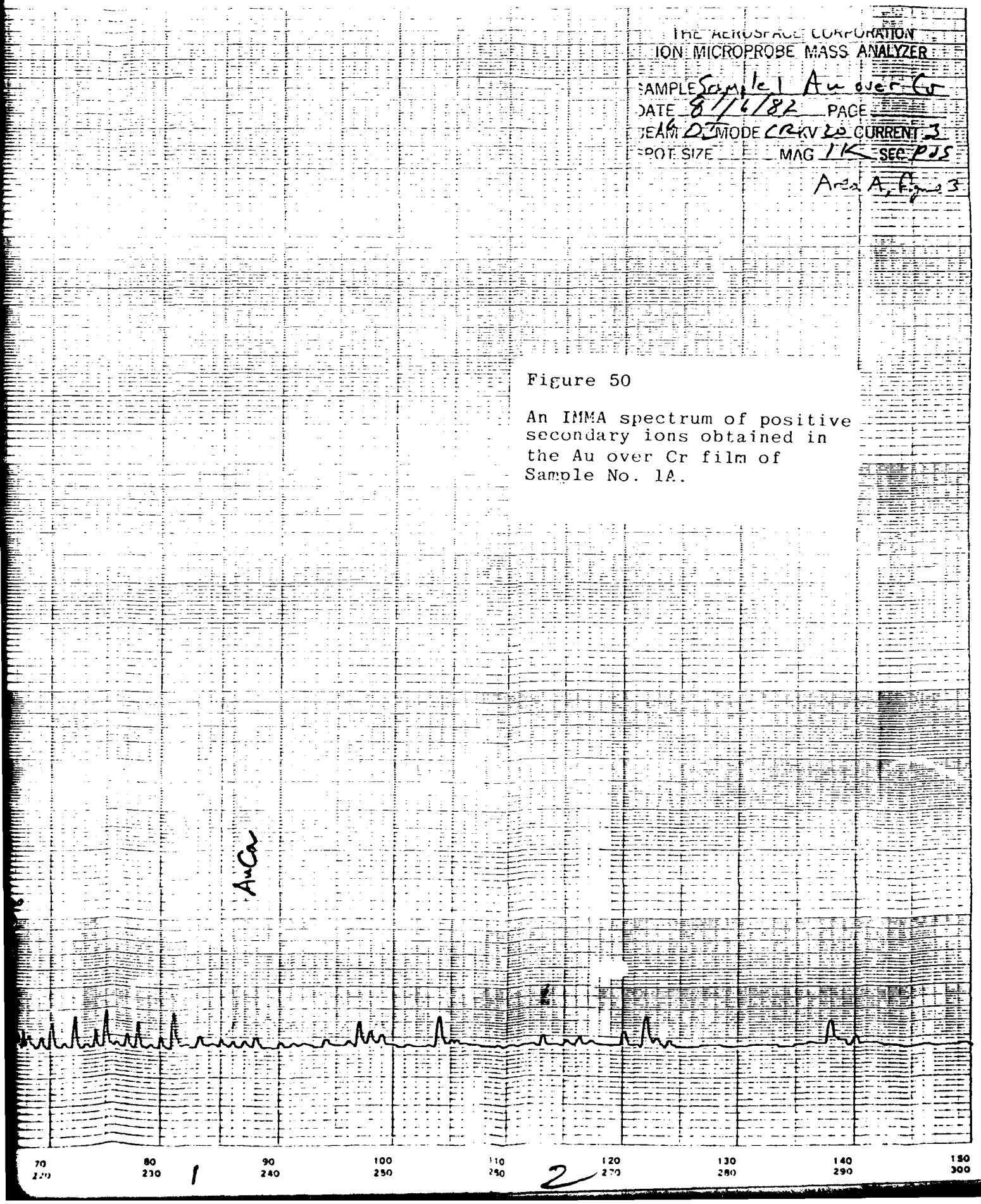
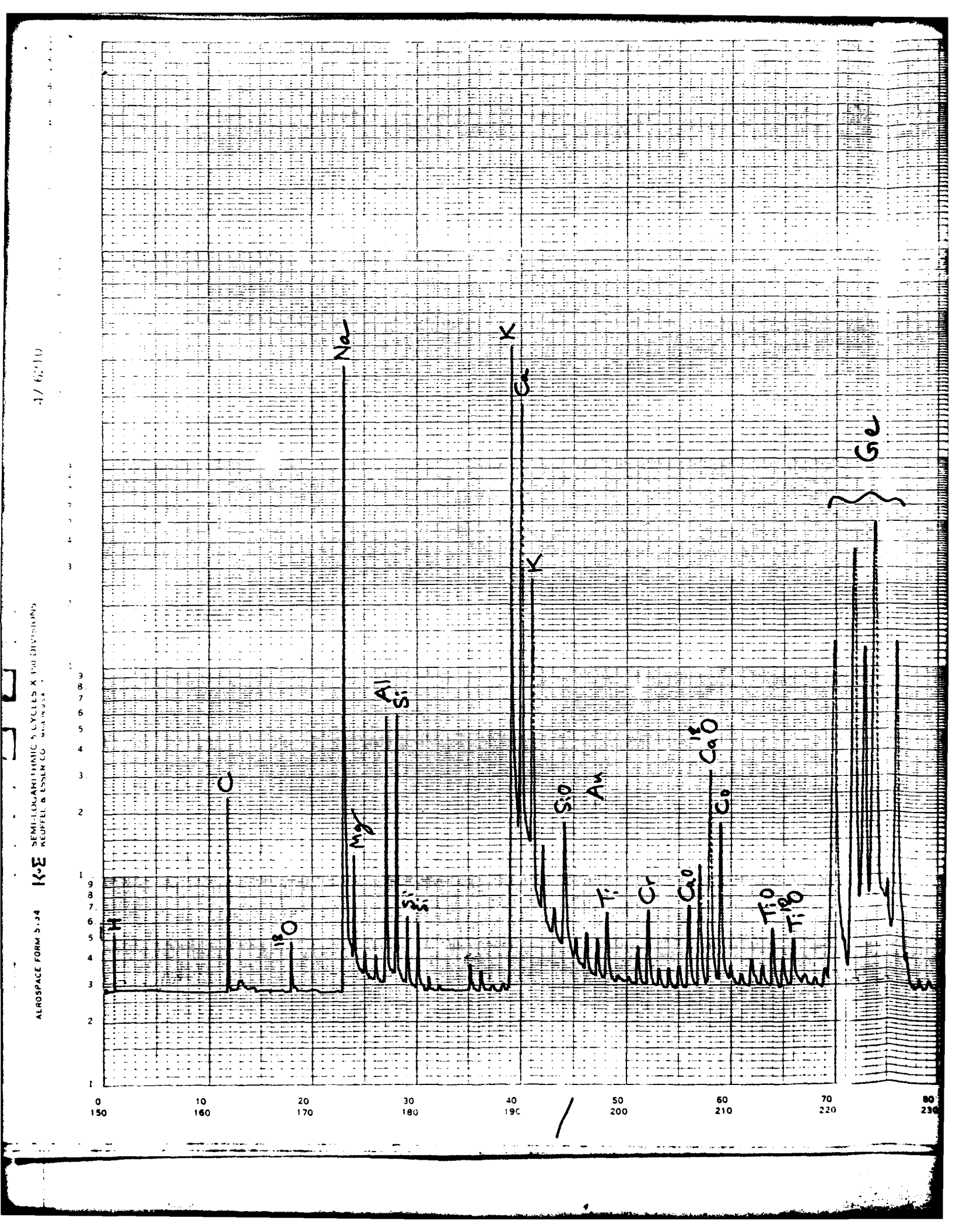


Figure 50

An IMMA spectrum of positive secondary ions obtained in the Au over Cr film of Sample No. 1A.

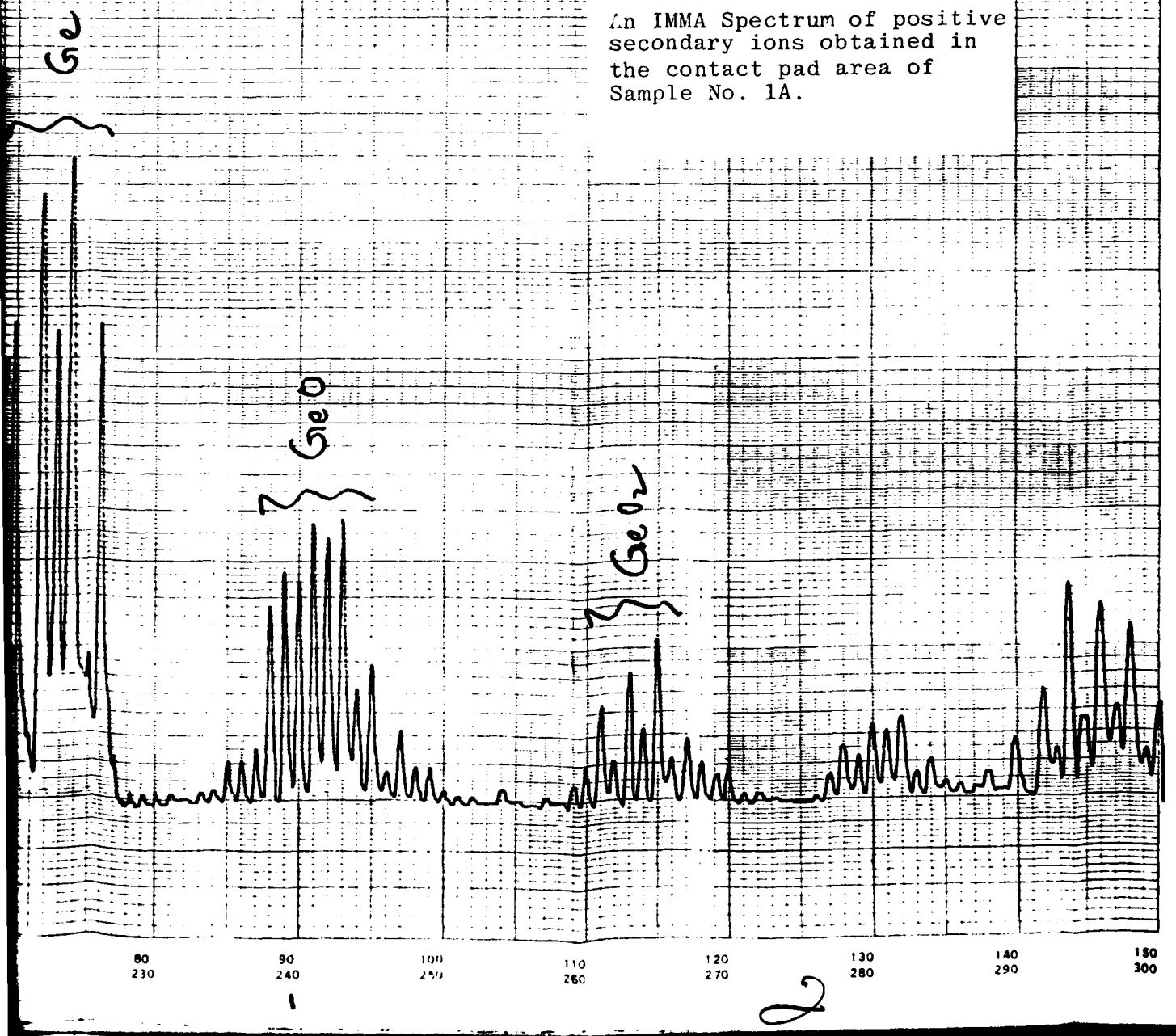

H
C
18O
Na
Mg
Al
Si
Si
K
Ca
K
Si O
Ar
Ti
Cr
Ca O
Ca
Ca O
Ti O
Ti 80
Ga
0
10
20
30
40
50
60
70
80
150
160
170
180
190
200
210
220
230

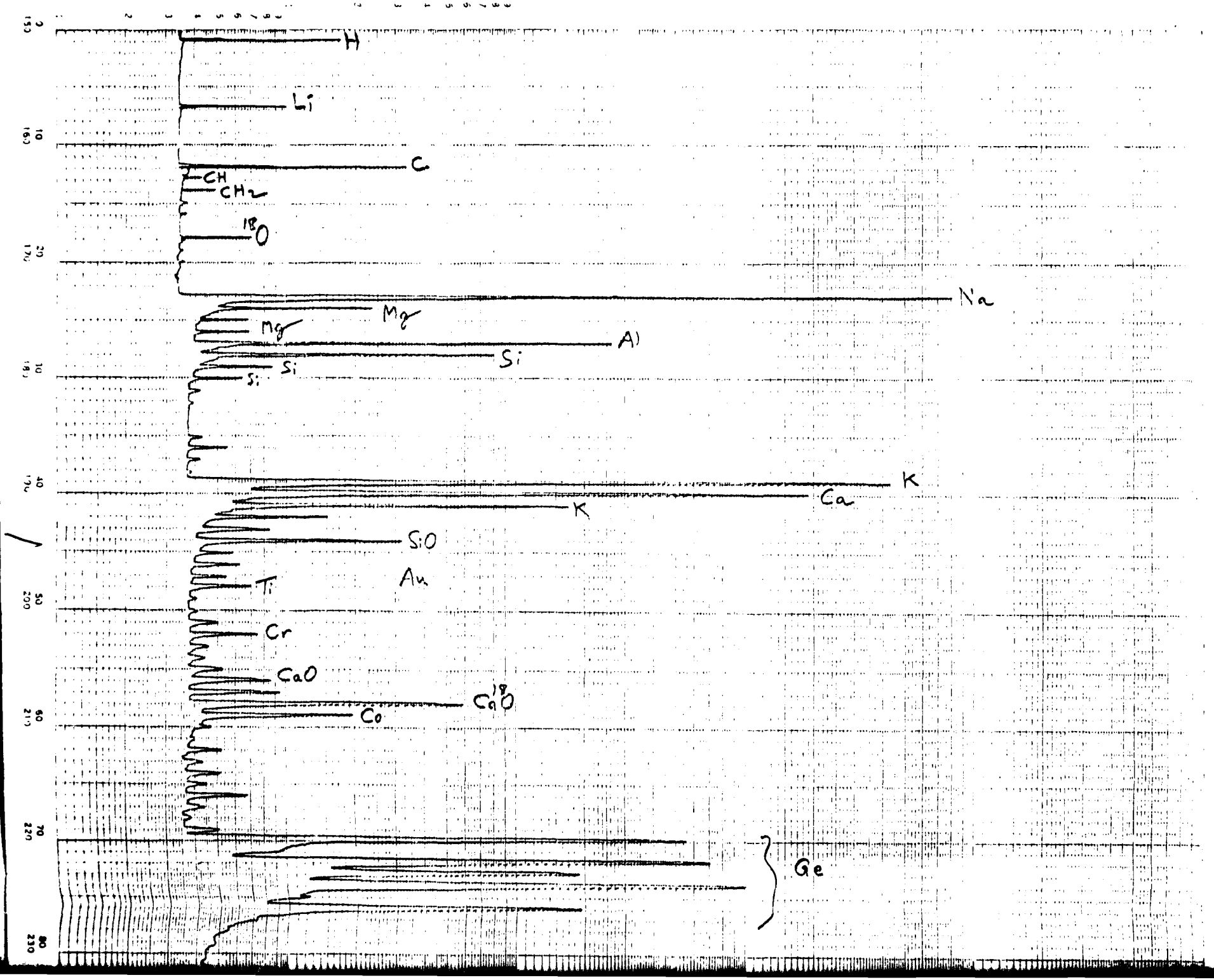
THE AEROSPACE CORPORATION
ION MICROPROBE MASS ANALYZER

SAMPLE #1 Contact Pad Area
DATE 8/16/81 PAGE 1
BEAM D+ MODE S-R KV 2 CURRENT 3
SPOT SIZE ____ MAG 11C SEC POS
Area B, Figure 3

Figure 51.

An IMMA Spectrum of positive secondary ions obtained in the contact pad area of Sample No. 1A.




H
Li
C
CH
CH2
18O
Na
Mg
Mg
Al
Si
Si
Si
K
Ca
K
SiO
Au
Ti
Cr
CaO
Ca18O
Co
Ge

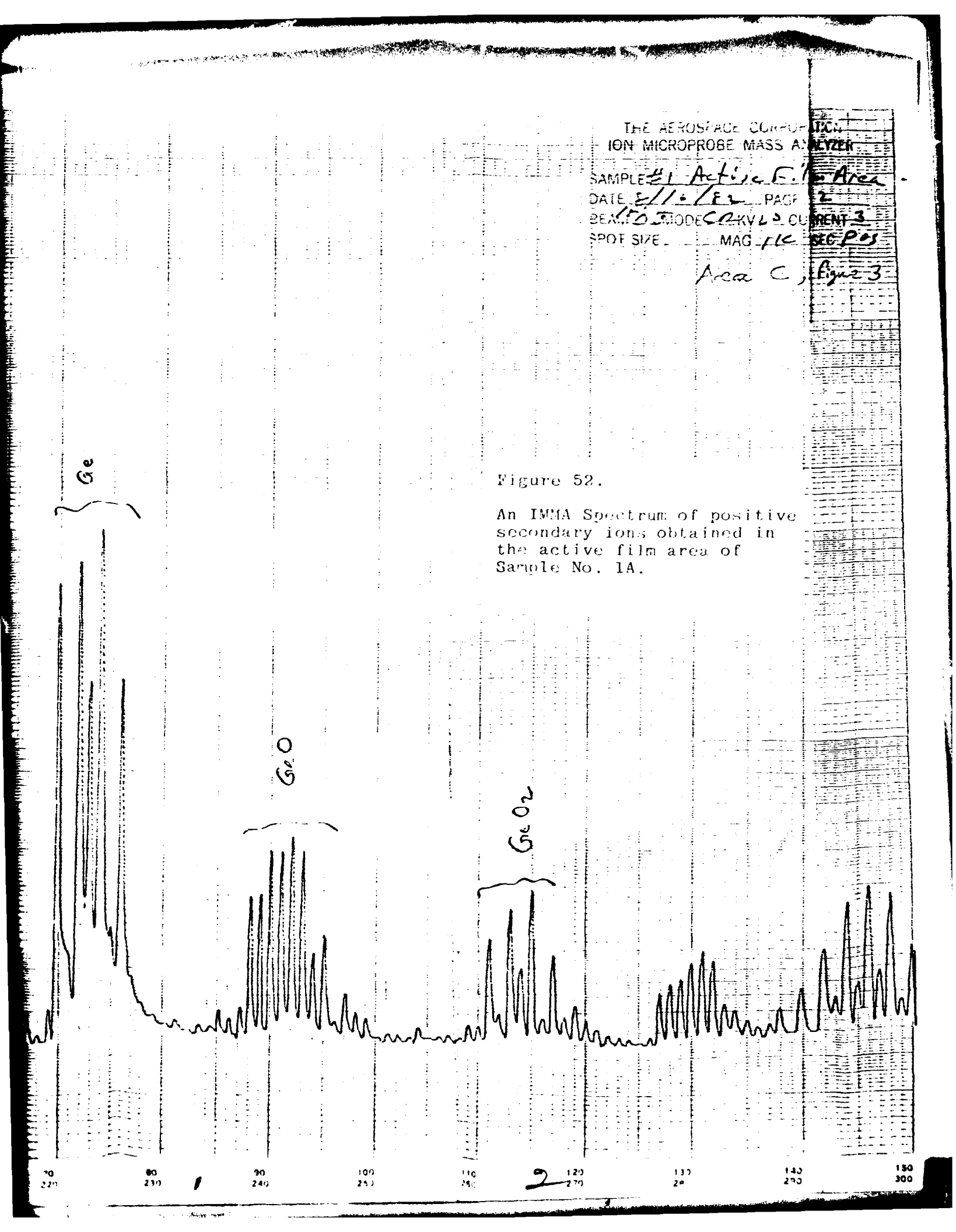


Figure 52.

An IMMA Spectrum of positive secondary ions obtained in the active film area of Sample No. 1A.

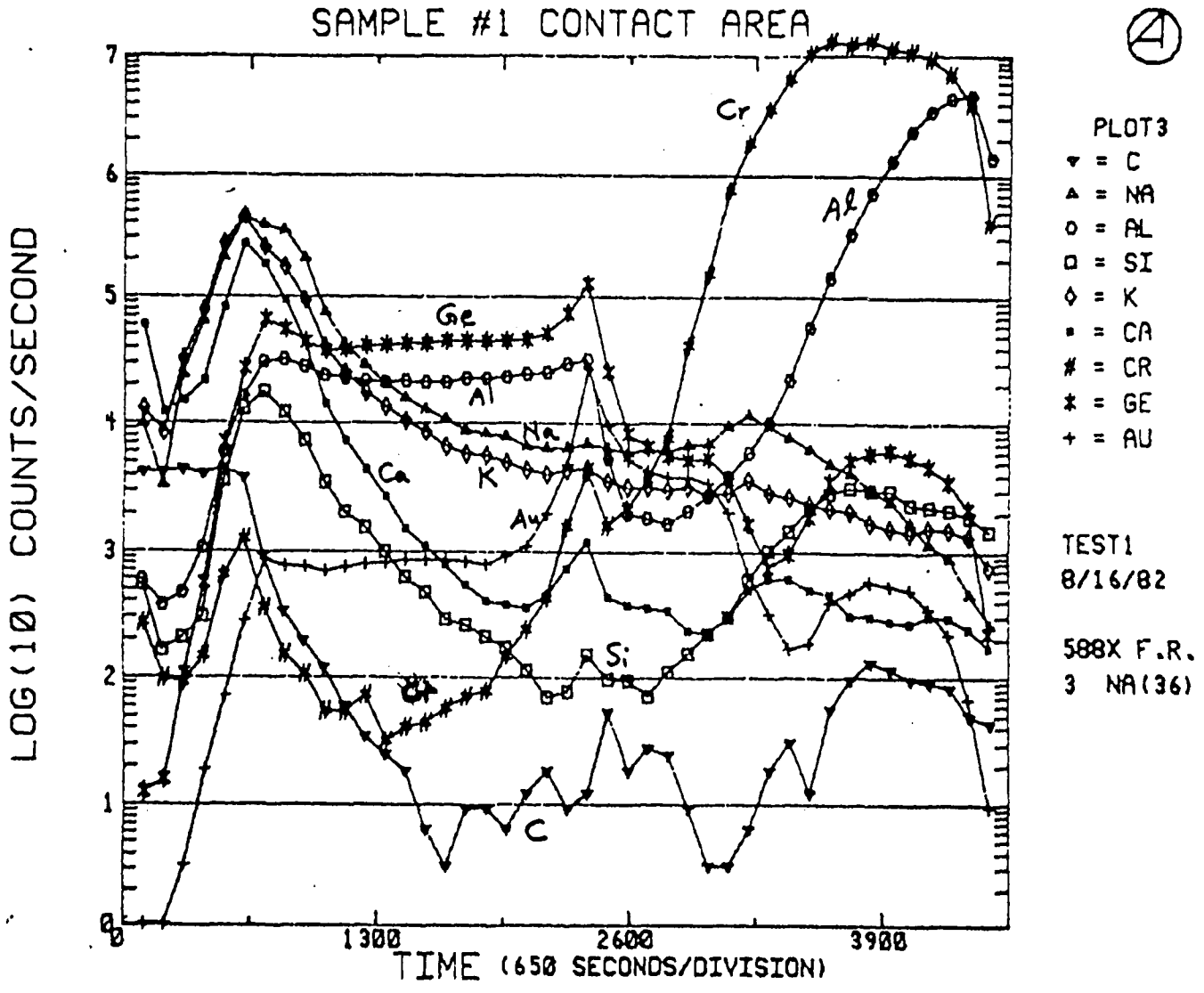
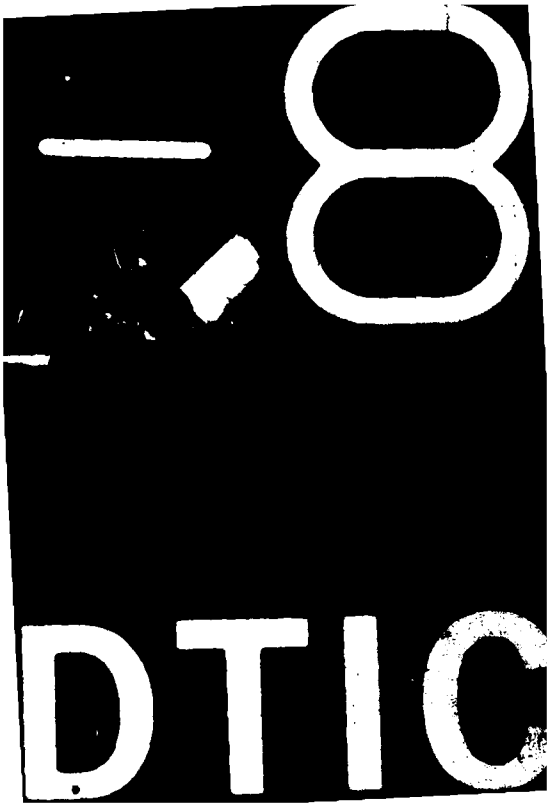


Figure 53. An IMMA depth profile obtained in the contact pad area of Sample No. 1A.


-8
DTIC